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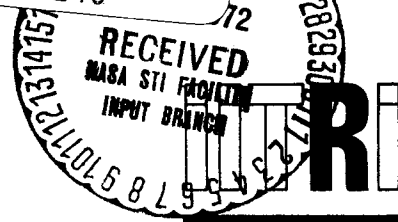
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FOR UNATTENDED SATELLITE TAPE RECORDERS  
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FIVE YEAR MAGNETIC TAPE FOR  
UNATTENDED SATELLITE TAPE RECORDERS

IITRI Project No. E6205  
Phase I Final Report  
Contract No. NAS5-21623

National Aeronautics and Space  
Administration  
Goddard Space Flight Center  
Greenbelt, Maryland

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February 1972

IIT RESEARCH INSTITUTE

## FOREWORD

This is the Phase I Final Report on IIT Research Institute Project No. E6205 entitled, "Five Year Magnetic Tape for Unattended Satellite Tape Recorders." The work was performed for the National Aeronautics and Space Administration, Goddard Space Flight Center, under Contract No. NAS5-21623.

The work completed over a nine month period was monitored by Mr. Carl Powell of Goddard Space Flight Center. IITRI personnel contributing to the technical content of this report include G. A. Zerlaut, K. Gutfreund, Dr. C. Giori, I. Lesevicius, J. Brzuskiwicz, and P. Mencinkas of the Chemistry Division; and R. J. Owen, G.S.L. Benn, L. B. Townsend, R. C. Gortowski, and J. Kurinsky of the Electronics Division. Fiscal and administrative supervision was directed by G. T. Jacobi, Assistant Director, Electronics Division.

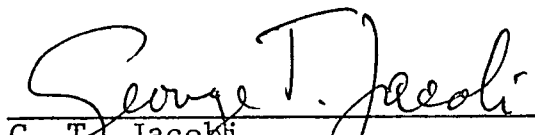
Respectfully submitted,

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## ABSTRACT

The requirement that a data storage magnetic tape recorder shall reliably operate unattended in space over a period of five years has highlighted the need for a magnetic tape structured to these exacting requirements. The main objective of this program is therefore the development and fabrication of a quantity of long life magnetic tape with properties selected specifically for unattended operation in spacecraft tape recorders.

The prime objectives of the first phase effort have been the establishment of the tape performance criteria necessary to achieve the life requirement and the identification of four generically different polymer binder candidates capable of fulfilling these criteria.

A detailed analytical consideration of various binder systems was undertaken. This included the chemical aspects of the binders, cohesion and adhesion effects, stability and the mechanical and physical properties. An initial selection of several binder resins were then examined in more detail together with a variety of other coating constituents. Numerous coatings were formulated and prepared as unsupported films independent of the carrier base film. The ability to form free films of these polymers and their combination with various oxide loadings and other additives allowed a rapid selection of four polymer candidates for a five year magnetic tape.

These four polymers were then extensively analyzed, particular attention being paid to their behavior in areas that were known to be life limiting, such as friction properties and thermo-adhesive properties. Short length tape samples were then fabricated in pilot quantities. These samples were evaluated under actual running conditions which included physical, magnetic, and extensive life testing. The ability of these sample tapes to withstand 50,000 bidirectional tape passes under fairly harsh operating conditions indicated that the preliminary analyses and selection criteria established during this program are sound.

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FIVE YEAR MAGNETIC TAPE FOR UNATTENDED  
SATELLITE TAPE RECORDERS

1. INTRODUCTION

This is the final report on the effort undertaken during Phase I of a four phase program under National Aeronautics and Space Administration, Goddard Space Flight Center, Contract No. NAS5-21623, IITRI Project No. E6205, entitled "Five Year Magnetic Tape for Unattended Satellite Tape Recorders."

1.1 Background

The National Aeronautics and Space Administration and numerous other organizations have experienced considerable difficulty in determining an appropriate magnetic tape type for use in reliable, unattended, magnetic tape recorders. For several reasons, including the low volume of tape procured for spacecraft applications and the proprietary binder systems resulting from the highly competitive nature of the tape manufacturing industry, a tape user is normally faced with the necessity of procuring virtually an unknown item by brand designation only. The situation of having to employ a vital component aboard a flight system with very little data for the specification or qualification of the product itself is not consistent with NASA philosophy of component and subsystem testing to improve reliability.

The choice of a particular tape type in unattended satellite recorder applications is of prime importance to insure utmost reliability during the mission life. In the past, the choice of a magnetic tape type has been achieved only by laborious testing techniques and its selection was made from several varieties of magnetic tapes currently produced for either the computer or instrumentation fields. The selection from existing tape types was imposed simply because the small demand for satellite applications has not stimulated

the manufacturer to design or fabricate a special tape specifically for this limited use.

Even when a tape type has been selected from this large field it is still necessary to individually test each reel of tape prior to use as experience has shown that not only do the same tapes vary from batch to batch, but even from reel to reel. To circumvent these changes, users have been known to purchase entire webs of one particular tape type in an attempt to obtain some degree of uniformity over the long term.

Problems have also been encountered when a manufacturer very slightly changes the formulation of a tape type but does not change the type designation. Such changes may be made to improve the quality for the tapes prime use (i.e., computer or instrumentation applications) but the change may seriously influence the character of the tape being used in a satellite recorder. Even more catastrophic to the limited user, is when a well proven tape type is removed from the market and additional supplies are severely restricted or non-existent.

The limited user is therefore faced with the problem of having to purchase a vital component for his system by manufacturers type number only with little or no knowledge of the exact specification or character of the product itself.

Until recently, the complexity of magnetic tape has restricted the specifications to those of the more simple physical and magnetic properties only. Recent studies have revealed that these limited specifications, although suitable for general use, are inadequate in characterizing the tape type when a specialized use is required as in satellite recording applications. An example of a normally unknown property which seriously affects the performance in satellite applications is the presence or lack of chlorine in the tape. This is rarely specified, but the presence of chlorine should restrict the use of the tape, especially in applications where

the tape remains in stationary contact with the head for long periods of time.

This is one of many properties which can have serious implications on the overall reliability of unattended satellite recorders even for one year continuous operation. The extended operation of orbiting satellites in current and projected missions placed an even greater burden on maintaining the integrity of the magnetic tape during mission life. It is for this reason that the development of a magnetic tape where all of the considerations were made towards life was undertaken.

## 1.2 Program Objectives

The necessary requirement that a magnetic tape recorder will operate reliably for unattended operation in space over a period of five years has highlighted the need for a magnetic tape to be structured to these exacting requirements. It is therefore for this reason that the prime objective of this program is the development and fabrication of a quantity of long life magnetic tape with properties selected specifically for unattended operation in spacecraft tape recorders. Coupled with the life requirement of a minimum of 50,000 cycles (two directions per cycle, reel to reel) the magnetic tape has to be capable of performing in accordance with all electrical and mechanical requirements, specifications, and constraints documented in NASA/GSFC specification No. S713-P-112.

To achieve the overall objective of this program the effort has been defined into four distinct phases:

- |           |  |
|-----------|--|
| Phase I   | Selection of Polymeric Binder Systems and Tape Performance Criteria. |
| Phase II  | Optimization of Coating Systems.                                     |
| Phase III | Refinement of Magnetic Tape.   |
| Phase IV  | Delivery of Product and Final Report.                                |

This document is the final report on the effort undertaken during Phase I of this program.

### 1.3 Phase I Objectives

The primary objectives during the first phase effort were the establishment of the tape performance criteria necessary to achieve the life requirement and the identification of four generically different polymer binder candidates capable of fulfilling these criteria. The emphasis, during Phase I, on the choice of these polymer binder candidates relates to the criticality of this tape constituent to the failure modes identified during recent studies (NAS5-11622). These failure modes include abnormal head to tape friction, adhesive interaction between the tape and recording heads and severe oxide shedding.

### 1.4 Report Organization

Section 2 of this report is a brief outline of the guidelines used for the current study. This is followed with Section 3 where the analytical considerations of binder systems are outlined. This includes the chemical aspects of the binder, its cohesion and adhesive behavior, the importance of the binders stability and mechanical and physical properties and finally the initial selection of binder polymer candidates. Section 4, entitled Experimental Investigations, details the various binder resins and coating constituents used during this study. Methods of formulation and preparation are given in detail, as are the measuring techniques that have been adopted. This is followed by the results which are discussed.

Section 5, entitled Preliminary Assessment of Tape Performance, is devoted to the tests undertaken on the short length samples of various polymer binders fabricated on a pilot coating apparatus at IITRI. It briefly describes the test methods and tabulates the results obtained on seven

different tape samples. Included in this evaluation were the IITRI Guideline Tests formulated under NASA Contract NAS5-11622, measurement of the frictional properties of the tape samples, magnetic properties of the coatings, and extended life tests under specific environmental conditions. The report concludes with a summary of the Phase I efforts and outlines a logical progression into the second phase of the program.

## 2. GUIDELINES FOR CURRENT STUDY

Considerations of magnetic tape properties for conventional tape use (Ref. 1) as well as careful evaluation of the requirements imposed on instrumentation tape for satellite recorders, based on results obtained in a previous investigation at IITRI (Ref. 2), provided the needed guidelines for the current study. The importance of the nature of the polymer binder as well as of the coating composition variables were found, from these considerations, to assume paramount significance in the selection of a suitable binder for high performance instrumentation magnetic tape for satellite recorders. Since it was realized that the reliability of magnetic tape in spaceborn satellite recorders depended on many factors, some of which were mutually interfering, the choice of the primary coating constituent had to be viewed critically in the perspective of stringent requirements imposed on the tape as a whole. These requirements called in general for a minimum of head/tape friction, freedom from all types of adhesive interaction, and sufficient coating integrity to eliminate the undesirable phenomenon of oxide shedding. In translating these requirements into polymer structure-related properties, it became apparent that one had to fully appreciate the fact that the binder represented a component in the coating system, whose performance could greatly, and not always predictably, be modified by other system ingredients.

Thus, the selection of suitable binder systems had to be considered not only from the viewpoint of apparent merits of candidate polymers, but immediate attention had also to be given to the effects of chemical and structural variables of the binder on the properties of the coating system. These properties included the degree of accommodation of gamma iron oxide, cohesiveness of the pigment-binder system, its compatibility with electrical conductivity-promoting agents (carbon), friction-reducing compounds, and dispersing agents.

### 3. ANALYTICAL CONSIDERATIONS OF BINDER SYSTEMS

In approaching the problem of selecting a suitable polymer binder for instrumentation magnetic tape for satellite applications, consideration has been given to the nature of some commercially used binder systems and their properties with regard to structural integrity, adhesion behavior, thermal stability and other physical characteristics. The properties of these polymers are discussed in the following sections.

#### 3.1 Chemical Aspects of Binders

The purpose of a binder is to provide a cohesive matrix for particulate magnetic materials to impart to the coating structural integrity and permit firm bonding of the coating to a tape substrate. This is generally accomplished by polymers of which many types are used for the dispersion of magnetic pigments. Some polymers, such as cellulose esters and ethers have been completely phased out from the manufacture of instrumentation tape coatings. Others, specifically butadiene-styrene polymers, vinyl chloride/vinyl acetate copolymers and vinylidene chloride/acrylonitrile copolymers have found limited use. Several instrumentation tape manufacturers appear to favor polyurethane-based binder systems, partly because of the relative thermal stability of this polymer, its castability from solution as a coating, and receptivity of pigments, in particular the magnetic iron-oxide. Epoxy and polyamide-modified magnetic tape binders have also been employed in some commercial tape coatings as indicated from measurements performed during the Magnetic Head/Tape Interface Study at IITRI (Ref. 2).

Other binder systems reported throughout the literature, but not widely used in commercial applications include:

1. A mixture of polyurethane resin and copolymer of vinylidene chloride and acrylonitrile (Ref. 3).
2. Polyalkylene-arylene ester glycols, linear dihydroxy terminated polymers and dihydroxy-terminated polyester amides, used for the modification of diisocyanate elastomer (Ref. 4).



3. Cellulose acetate and a condensation product of formaldehyde and toluene ethylsulfonamide (Ref. 5).
4. Melamine formaldehyde and polyurethane (Ref. 6).
5. Aryl isocyanate and acrylonitrile (Ref. 7).

All of these binder systems have been developed in an attempt to improve certain parameters such as the cohesion, adhesion, mechanical stability, and other physical properties. These properties deserve particular attention in the development of durable magnetic tape coatings.

### 3.2 Cohesion

The integrity of a binder depends on molecular cohesion, and the latter is closely associated with the chemical nature of the polymer. Unsatisfactory cohesion of a high-molecular-weight material can lead to mechanical failure at a low level of distortional stress work (Ref. 8). Failure at low levels of stress energy usually occurs in materials that either undergo excessive plastic deformation or rupture prematurely as a result of extreme brittleness. Obviously, both effects must be avoided in the development and formulation of high performance magnetic tape coatings. Toward this end, attention should be given to the use of plasticizers in relatively small amounts and the limitation of the quantity of inert additives (e.g., lubricants) that could disrupt the integrity of the binder. Also, the use of extremely rigid (highly cross-linked) resins should be avoided to maintain the distortional stress energy required to produce failure of the binder above the operational level associated with the transport of tape on the recorder.

Another factor that deserves attention with regard to the integrity of magnetic coatings is cohesion of the pigment-binder system. Actually, adhesion effects between pigment and binder are also important here, although in those instances where the interaction between filler and resin is relatively small, e.g., in inert additive-filled polyurethane elastomers (Ref. 9) many time-dependent properties remain qualitatively similar for the filled as well as unfilled polymer. The high-

modulus material dispersed in the amorphous phase of the resin does not undergo deformation under stress, but the surrounding elastomer detaches from the particles forming vacuoles around them. If the deformation of the polymer is reversible within the operational stress range, the partially detached particles return to the initial position in the resin when the stress is relieved. However, should the elastic limit of the resin be exceeded, the voids formed around the filler particles would remain as permanent vacuoles. This would not only weaken the pigment-binder system after successive traverses of the tape across the record/playback heads, but would release embedded filler particles from the resin, thereby contributing to excessive debris formation. It is apparent from the foregoing considerations, that cohesion of the binder and integrity of the pigment binder system require optimization to ensure satisfactory tape performance over an extended period of use.

### 3.3 Adhesion

The adhesive behavior of magnetic tape binders is important in the overall performance of magnetic tape since it relates directly to (1) pigment-binder interaction, (2) adherence of magnetic coating to tape substrate, and (3) "sticktion" of tape-to-head surfaces. For these reasons, the phenomenon of adhesion deserves close attention.

The adhesion of polymers, such as represented in organic binder systems of magnetic coatings, to solids is often considered a result of van der Waals interactions, although other forces, notably ion-dipole (Ref. 10), ion-ion (Ref. 11), and hydrogen bond (Ref. 12, 13) have been considered responsible for the adhesion of resins to metal and other surfaces. Poor contact on a molecular level minimizes the interaction (adhesion) of an organic compound with a solid surface by London forces, since the dispersion force field has an extremely limited range (it is proportional to the seventh power of distance). The requirement for intimate contact between adherend and adhesive is less stringent for systems in which the interactions are of the dipole-dipole or electrostatic nature. Such interactions are believed

to take place at metal surfaces. It has been emphasized that good adhesive bonding of hydroxy- or carboxy-functional resins to metals is accomplished through interaction of these groups with the oxygen on the metal (Ref. 14). The interaction of hydroxyl group-containing surfaces with carboxy-functional compounds was postulated to take place through hydrogen bonding which was considered a special case of electrostatic interaction (interaction of proton donor and acceptor) in studies of the adhesion of polymethacrylic acid and methacrylamide derivatives to glass surfaces (Ref. 15). Although the formation of primary chemical bonds between substrate and polymer is not considered necessary for the formation of strong adhesive joints, the existence of such bonds has been demonstrated in desorption studies of vinyl-trichlorosilane-treated glass surfaces (Ref. 16). Also, bonding of sulfur-containing natural rubber to copper is believed to proceed through formation of a chemical linkage (electrovalent bond) at the metal/rubber interface.

The specific interaction of polymers at reactive sites on solid surfaces and formation of adhesive bonds has been postulated in studies of the adhesion of diisocyanates to metals (Ref. 17). Here, the formation of a chemical bond between free isocyanate and hydrated metal-oxide groups was considered responsible for the good adhesion of polymerized hexamethylene diisocyanate to steel, light metal alloys, porcelain and other substrates. In epoxide polymers, the ethylene oxide of hydroxyl functionality is credited with the adhesive capacity of these polymers in metal or ceramic bonding applications.

The relationships discussed above suggest the desirability of selecting those binders for high performance instrumentation magnetic tapes which, after curing, would contain a minimum of free functional groups capable of posing "sticktion" problems by virtue of their potential interaction with recording head surfaces. However, prior to the final curing stage, the presence of reactive sites on the polymer or partially polymerized monomer is desired to promote adhesion of the magnetic coating to the tape substrate, enhance the binding of pigment particles

in the matrix, and aid the process of binder polymerization to obtain a high-molecular-weight material from the film-forming vehicle.

### 3.4 Stability of Binder

The organic polymer which comprises the matrix for magnetic oxide particles in recording tape should satisfy the requirement of thermal stability, since frictional contact between the coating and head surface can produce local heating effects that might lead to the degradation of the polymer into low molecular-weight fragments. These fragments, adhesively bonded to the head, may not only produce excessive head/tape separation and thereby result in the dropout of information, but they can also promote head/tape interaction at the active sites of the degraded product. This interaction can be enhanced by the diffusion of incompletely polymerized binder constituents to the tape surface. Low molecular-weight constituents can be found in the binder matrix, if the polymerization or curing of the organic material were impeded by the presence of excessive amounts of additives, including nonreactive lubricants. For these reasons, it appears desirable that the binder have good thermal properties and exhibit a tolerance for essential additives. The number of these additives should, if possible, be reduced to minimize their diffusive transfer and exudation to the surface. Polyurethanes represent polymers with reasonably good thermal characteristics (melting point 120 to 250°C, depending on the nature of the diisocyanate and diol), but, under unsatisfactory polymerization conditions, they may contain low molecular-weight hydroxy- and aminofunctional fragments that could be adhesively transferred to the head surface thus increasing the probability of head/tape adhesion. Thus, in order to minimize the degradation of the polymer in sliding friction and reduce the danger of the formation of low molecular-weight fragments, the binder should have maximum thermal stability inasmuch as this stability is consistent with other required binder characteristics.

### 3.5 Mechanical and Physical Properties

The ultimate mechanical behavior of polymeric materials, as reflected in their tensile strength, elongation and toughness, determines in large measure the performance characteristics of resins. These properties are also important for magnetic tape binder systems. Plasticized polymers, particularly those of lower molecular weight are characterized by a low-yield strength (the stress at the transition from elastic to plastic range where permanent deformation takes place). Because of their inherently low strength, these polymers would not qualify for use in instrumentation tape binders.

On the other hand, high strength alone does not provide the criterion of binder acceptance if concomitant low elongation would impart pronounced brittleness to the resin, thus emphasizing its susceptibility to failure at small strains. The capability of the binder to undergo small, reversible deformations must be ascertained to ensure the integrity of the coating during the traverse of magnetic tape across the recording head surface at prevailing wrap angle conditions. To illustrate the differences in strength-elongation properties of film-forming resins that include polymers used in binder systems, the tensile strength and ultimate elongation of these resins are shown in Table 1.

Table 1

#### MECHANICAL PROPERTIES OF HIGH-MOLECULAR-WEIGHT RESINS

<u>Resin</u>	<u>Tensile Strength (psi)</u>	<u>Elongation (per cent)</u>
Vinyl chloride/ acetate	2,500 - 5,000	100 - 500
Polyurethane elastomer	5,000 - 10,000	450 - 550
Polyamide	15,000 - 20,000	250 - 550
Polyimide	25,000	70

Another material characteristic associated with the performance of a binder in magnetic tape coatings is hardness. Hardness represents a combination of properties rather than a single material characteristic. It is sometimes expressed in terms of the minimum stress that is required to fracture the surface of a substance, for instance in scratch resistance measurements, or it can also be represented by the reversible or irreversible deformation of a surface by forces operating in a compressive mode of action. In the latter case, hardness is defined as the resistance of a material to deformation. In this connotation, stress-strain and modulus of elasticity properties could also be used to characterize the resistance of a tape binder to damage by the asperities of a recording head surface and the mechanical properties reflect in some measure the sensitivity of the binder toward indentation by the asperities. It would then follow, that a polymer with a high modulus of elasticity should be preferred for binder applications in durable magnetic coatings provided that other binder requirements were satisfied. This assumption is not rigorously valid, since it postulates the equivalence of the deformation behavior of a polymer in compression and tension.

### 3.6 Initial Selection of Binder Polymers

The foregoing considerations delineate the operating latitude within which the selection of candidate binder polymers for a durable instrumentation magnetic tape is possible. Thus the polyolefins such as polyethylene, polypropylene, polybutenes, and various ethylene copolymers cannot be considered for application in the subject project since poor thermal stability, relative softness, and low shear strength disqualify these polymers from use in shear and friction-sensitive coating applications. Similarly, diene resins (natural rubber, polybutadiene, polyisoprene, and some of their copolymers with isobutylene, or styrene) are not well suited for the intended use in this program because of their adverse frictional characteristics, residual adhesion, and unsatisfactory thermal properties. Aliphatic vinyl polymers including polyvinyl chloride, polyvinyl acetate, polyvinylidene

chloride and allyl polymers, some of which have found application in binders for audio-tape magnetic coatings, would be disqualified because of thermal stability considerations and possible adverse effects of polymer degradation products, notably HCl. Similarly, acrylic polymers (polymethyl acrylate, polybutyl acrylate, polymethyl methacrylate and polyacrylamide) used extensively as binders in the paint industry, do not possess the thermal and mechanical stability characteristics under shear and friction conditions.

On the basis of the previously discussed mechanical and thermal considerations, a series of potentially suitable, pigment-receptive polymers has been selected for investigation of their applicability in instrumentation tapes for satellite recorders. These polymers included linear amide, imide, carbonate and siloxane resins as well as cross-linked polyurethanes and polyepoxides. Some of the polymers mentioned above, notably the urethanes and epoxides, have found application in commercial instrumentation tapes. The manufacturing demands inherent in the volume production of these tapes unquestionably imposed processing requirements (coating, curing, and drying) that necessitated the use of specific coating ingredients and coating conditions. The performance of these tapes under normal conditions could be considered satisfactory even if the properties of the coating with regard to inherent strength, pigment-binding capacity and binder inertness (in cured state) were not fully optimized. As these conditions no longer apply to instrumentation tape for satellite recorders, the selection of a binder for this tape demands more attention to the performance aspects of the recording medium than to volume production requirements. Process limitations, even if solvable by methods that could be considered inexpedient in commercial production, thus assumed secondary importance to material requirements. Accordingly, attention was directed toward the effects of polymer and formulation variables on the properties of magnetic coatings, while less emphasis was given to processing conditions.

#### 4. EXPERIMENTAL INVESTIGATIONS

The considerable advantages derived from the investigation of laboratory-prepared coating formulations rather than the magnetic coatings of commercial tapes, lies not only in the precise knowledge of the coating ingredients, but also in the possibility of determining performance-related properties from measurements on unsupported films of these coatings. The difficulty of removing the magnetic medium from commercial instrumentation tape necessitated the use of indirect and rather cumbersome test methods for the evaluation of tapes in a previous study conducted at IITRI (Ref. 18). In the current investigation, the availability of unsupported films of coating formulations prepared in the laboratory greatly increased the effectiveness of binder evaluation efforts.

##### 4.1 Binder Resins

In accordance with the guidelines developed for the selection of binders for instrumentation tape coatings, attention was given to epoxide, urethane, siloxane, polyamide and polyimide resins. Also, in a parallel study, a carbonate-amide polymer with potentially good pigment-binder properties and thermal characteristics was synthesized in a reaction involving bisphenolamide and phosgene.

##### 4.1.1 Epoxides

The desirability of determining the effects of chemical variables (for a particular polymer type) on performance-related properties of formulated magnetic coatings, suggested the investigation of aromatic and aliphatic analogs of epoxide resins. Consequently, a high molecular-weight aromatic epoxide resin, DER-669 (Dow Chemical Co.) and its aliphatic corollary, DER-736 (Dow Chemical Co.) were investigated in a comparative study, to ascertain the effects of structural variables on the mechanical, thermal, and frictional properties, as well as formulation (pigment-binding) characteristics of these resins. Methylene dianiline, DEH-50 (Dow Chemical Co.) was used as curing agent for the epoxides, while methyl-ethyl-ketone (MEK)



and xylene were employed as solvents to adjust the viscosity of pigment-containing systems to coating consistency. Since initial measurements indicated a pronounced brittleness of aromatic epoxide coatings, a polyamide, Versamid-115 (General Mills Co.), was used as a binder-modifying additive. This additive was employed in amounts ranging from 25 to 75% by weight of epoxide resin. The polyamide served in the modified epoxide binder as curing agent; unmodified aromatic and aliphatic epoxides were cured with DEH-50; which was used in an amount of 25% by weight of resin. The cure cycle for laboratory-prepared specimens involved heating at 55, 125 and 175°C for 16, 2 and 2 hrs, respectively. Polyamide-modified and epoxide-coated tape, prepared in pilot quantities on the laboratory coater, was subjected to a 5 hr cure at 75°C. In some instances, as indicated, the cure cycle varied from the above-given conditions.

#### 4.1.2 Urethanes

Polyester-based urethane binders were investigated in formulations prepared from an experimental, blocked isocyanate, E-320 (Mobay Chemical Co.), and a polyester resin, E-331 (Mobay Chemical Co.), whose thermal properties were reported by the manufacturer to be particularly desirable for the formulation of stable coatings. The binders contained 9.3p E-320, 15.4p E-331, and 11.2p Xylene: cellosolve acetate (1:1) solvent. The cure was effected by heating the resin without catalyst at 175°C for 30 min; with dibutyl tin catalyst (0.1%) a cure of 15 min at 150°C was required. Another urethane polymer, X-4510-40 (Cargill, Inc.), was obtained to determine its suitability in coating applications. This polymer, formulated with 60% xylene:cellosolve acetate (1:1), was cured by heating at 175°C for 30 min, during which time the blocking agent was volatilized.

#### 4.1.3 Modified Polysiloxane Binders

The applicability of a silicone binder was investigated in a study of a polyester-modified silicone resin, 6103-60 (Cargill, Inc.), which was credited with particularly good mechanical properties, and heat distortion characteristics. Films prepared

from this resin were cured at 175°C for 30 min, followed by a short exposure to 200°C. The pronounced flexibility and relatively low strength of the silicone film observed in initial tests suggested further modification of the resin by reaction with a rigid, thermally stable urethane polymer, X4510-40 (Cargill, Inc.). Clear and pigmented films, containing 25, 50 and 75% X-4510-40 with the balance provided by 6103-60, were formulated and cured by heating for 2 hrs at 125°C followed by 30 min at 175°C. Also, the 6103-60 silicone resin was used to flexibilize, without loss of thermal properties, the rigid, DER-669 polyepoxide.

#### 4.1.4 Polyamide Resin

In an effort to evaluate the performance of a thermally stable, and flexible resin in magnetic coatings, a tough polyamide, Elvamide 8061 (E.I. duPont deNemours & Co.), was used in several coating formulations. The resin was dissolved in a 70:30 methanol:trichloroethylene solvent system, to provide a 20% solution. However, the observed tendency of the dissolved resin to gel suggested the use of 5% benzyl alcohol in the solvent mixture, to prolong the shelf-life of coatings. Films of thus obtained coatings were heated at 40°C for 2 hr; residual solvent was removed by exposing the coating for 10 min in an air-circulating oven that was maintained at 120°C.

#### 4.1.5 Polyimide

The advantages derived from the exceptional thermal stability and good mechanical properties of polyimide resins in magnetic coating applications for instrumentation tape were investigated for a representative polyimide, DE 910-101 (de Beers Laboratories), coating system. The removal of solvent (xylene/dimethylformamide) and cure were effected by heating the coating in an oven at 50°C for 4 hr, and subsequently exposing it to 175 and 260°C for 4 hr and 15 min, respectively. It has been observed that, unless the coating received a full cure at 260°C, the specimens were extremely fragile. The importance of full cyclization of the imide by dehydration of the carboxylated intermediate became thus apparent.

#### 4.1.6 Amide-Carbonate Polymer

The amide-carbonate (AC) polymer that was synthesized at IITRI, according to the procedure discussed in the next section, was subjected to evaluation in coatings with regard to its mechanical properties, pigment-binding capability, thermal characteristics and the effects of molecular weight on these properties. The polymer obtained in lower molecular-weight fractions (inherent viscosity 0.19 to 0.3) exhibited poor structural integrity and unsatisfactory iron oxide-accommodating capability. However, polymers with an inherent viscosity above 0.5 produced coherent films when cast from dimethylacetamide solution at 20% solute content. The solvent was evaporated by heating for 12 hr at 50°C. Two polymer samples, one AC-2 with an inherent viscosity of 0.68, and another, AC-26 with an inherent viscosity of 0.65 were used in coating formulations.

### 4.2 Coating Constituents

#### 4.2.1 Pigment

Magnetic iron oxide (MO-2530), obtained from Charles Pfizer Company, comprised the pigment in coatings that were formulated with the previously mentioned binder systems. The amount of pigment used in the binder varied from 25 to 80% by total weight of coating (pigment-binder system). For most tape coatings, the upper pigment-loading level was maintained at 70%.

#### 4.2.2 Carbon Black

Carbon black powder of 425 Å (Std. Deviation = 250 Å) particle size, obtained from Shawinigan Co., was employed as a static electricity-reducing agent and lubricant in selected binder systems. This additive was incorporated in the binder after drying at 120°C for 12 hr, in amounts ranging from 3 to 5% by weight of resin. The 5% loading level appeared excessive, particularly in coatings that contained 70% iron oxide since; at this level, the structural integrity of the coating was considerably reduced. Therefore, in the preparation of tape in pilot quantities, the maximum level of carbon black was kept at 3% by weight of resin.

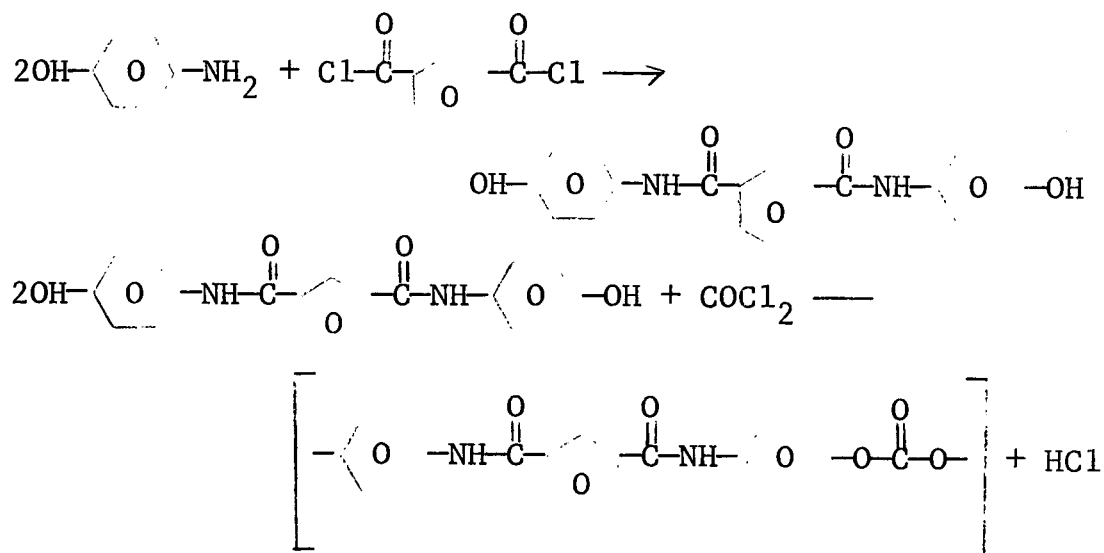
### 4.2.3 Other Additives

Although an attempt has been made to keep the number of coating ingredients to a minimum, in order to permit better evaluation of the properties and performance of binders, several additives have been used in the coating system. These additives included lubricants and friction-reducing compounds: a reactive silicone lubricant, SR-173 (General Electric Co.), heptafluorobutyramide (HFBA) and decafluorobiphenyl (DFBP). These additives were incorporated in the resin in amounts varying from 2 to 8%. In some binders, the use of 8% of HFBA greatly reduced the structural integrity of magnetic coatings that contained 70% iron oxide pigment.

### 4.3 Methods

#### 4.3.1 Synthesis of AC Polymer

The synthesis of the amide-carbonate (AC) polymer involved: (1) condensation of p-aminophenol with isophthaloylchloride (mole ratio 2/1) in pyridine solution, and (2) polymerization of the resulting bisphenoldiamide with phosgene. The reactions were carried out in the following way:



In a typical polymerization p-aminophenol (45.81g, 0.42 mole) is dissolved in dry pyridine (2,000 cc) in a flask equipped with nitrogen inlet, outlet and stirrer. The solution is cooled at 0°C and solid isophthaloylchloride (42.63g, 0.21 mole; previously recrystallized from n-hexane) is added under nitrogen with stirring. After stirring at 0°C for 4 hr, the solution is slowly heated to room temperature. Lithium chloride (140 g) is added to the solution. The solution is cooled again at 0°C and phosgene gas is added at a slow rate (0.03g/min) with stirring. During addition of phosgene the color of the solution turns orange, then gradually deep red. By continuing the addition of phosgene the color becomes lighter while the solution viscosity increases considerably. The polymer is then precipitated by pouring the solution into a large excess of water. The polymer material thus obtained is filtered, washed with water, methanol and chloroform, and dried at 80°C under vacuum. The yield is 70g (90%).

#### 4.3.2 Formulation of Coatings

Magnetic coatings, in 8-oz. test quantities, were prepared with different binder systems by milling in a ball mill a 10 to 20% solution of the resin in a suitable solvent, with the desired amount of iron oxide pigment (25 to 70%) and additives (carbon black, lubricant). The amount of solvent was adjusted in relation to the solids content and desired consistency of the coating. In polyamide-modified epoxide coatings, pigment, carbon black and additives were first milled with the solvent-diluted polyamide, before dissolved epoxide resin was added to the disperse system. In those instances where methylene dianiline was employed as curing agent, the solids were dispersed in a solvent-diluted epoxide solution, and the curing agent was introduced in the final phase of the milling process. Initially, coatings were milled for 48 hr, of which 2 to 4 hr were spent for dispersing the catalyst in the mixture. Later, as it became apparent that incomplete dispersion interfered with the deposition of uniform coatings on tape produced in pilot quantities, the milling time was extended to more than 100 hr.

#### 4.3.3 Preparation of Unsupported Films from Coatings

Free, unsupported films of binder polymers, with or without iron oxide pigment, were prepared by doctor-blade casting techniques on tin foil or Teflon release agent-treated Mylar substrate. The film was isolated from the substrate by peeling off the Mylar or by amalgamation of the tin foil. Thus, dry films ranging in thickness from 0.003 to 0.007 inch were obtained after curing. This procedure was used for all but polyimide coatings, since the latter required a curing temperature of 260°C that neither Mylar nor tin foil could sustain without damage. Consequently, a two-stage curing process was adopted in which tin foil-supported polyimide coatings were "B-staged" at 175°C, for 5 hr, after which the coatings, removed from the substrate by amalgamation, were placed on a Teflon sheet that permitted their final exposure to the 260°C curing condition. Unfortunately, this method was not always satisfactory, since the high temperature tended to distort the unattached films when they were placed in the oven on Teflon sheet. Better results were obtained when the polyimide coatings were deposited by doctor blade on Teflon release agent-pretreated Kapton (E.I. du Pont de Nemours & Co.) polyimide film. This thermally stable substrate permitted curing of the coating at 260°C, thereby eliminating the need of detaching and transferring the coating from tin foil to Teflon. After removal from the Kapton substrate, the cured films of coating material were cut in 1/2 x 6-in. strips on a twin-blade shear cutter. The thus obtained strips provided test specimens for the determination of mechanical properties of magnetic coatings.

#### 4.3.4 Preparation of Tape in Pilot Quantity

The preparation of magnetic tape in 12 to 25 ft lengths, for evaluation of its performance on the loop tester, necessitated the use of commercial coating equipment. Consequently, a laboratory bench coater (Custom Scientific Instruments, Inc.) has been assembled as shown in Figure 1. The 6-in.-wide Mylar tape web, placed between the rubber-covered drive roll and chrome finished doctor and coating rolls, was conveyed at a speed of 5 ft/min.

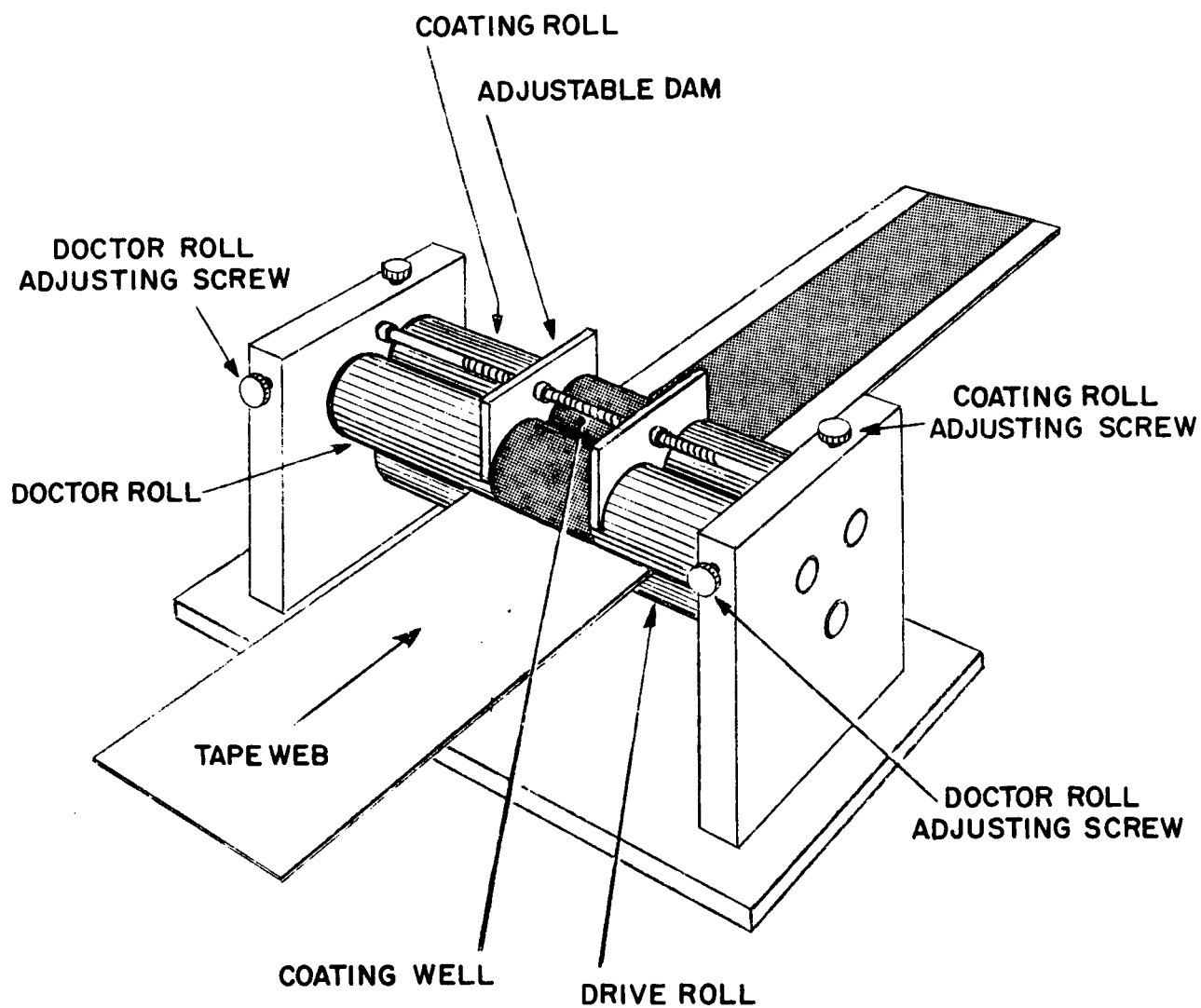


Fig. 1 LABORATORY BENCH COATER

Initial coating experiments were performed with polyamide (Versamide-115)-modified epoxy resin (DER-669, Dow Chemical Co.) at an iron oxide loading level of 70%. In order to confine the coating to a lesser area on the tape web, a Teflon dam with an adjustable span was mounted on the metal rolls. Preliminary coating trials did not produce satisfactory results, as the deposited coating had a streaked surface appearance as a result of rapid solvent evaporation and the replication of surface details of the not perfectly polished coating rolls on the deposited film. The use of a higher boiling solvent in the epoxy binder did not greatly improve the quality of the coating. However, this condition was greatly remedied when a doctor blade was used to meter out the coating in desired thickness.

The modified coating apparatus, shown in Figures 2 and 3, conveys the tape from an unwind reel, R, through a substrate (Mylar tape) cleaning pad, P, to a coating reservoir, C, which is mounted on a Teflon base plate, T. From here, the tape traverses a distance of approximately 2 ft to a thermostatically controlled heater rod, H, after having been provided with a coating layer of 0.5 mil wet thickness by the doctor blade, B. The coated, solvent-free tape is conveyed through Teflon guide rolls, G, to the uptake reel, U. Magnetic coatings of 0.0003-in. dry thickness have been deposited on Mylar and Kapton substrates by this method.

Since the laboratory coater accepted tape in 1-in. width, and the Mylar and Kapton substrates were obtained from the manufacturer (E.I. du Pont de Nemours, & Co.) in 6-in. rolls, a suitable slitter had to be designed. Such a slitter, utilizing 4 surgical blades as tools (Figure 4) permitted preparation of quadruplicate, 1-in. wide strips from the 6-in. web. Final slitting in which the coated tape was bilaterally trimmed to 1/2-in. width, was performed in an operation subsequent to coating. Using this method, 7 tapes, ranging in length from 12 to 25 ft. were prepared for evaluation of their properties on the loop tester. The magnetic coatings of these tapes comprised formulations based on modified epoxide resins, polyamides and amide-carbonate binders. The dispersed



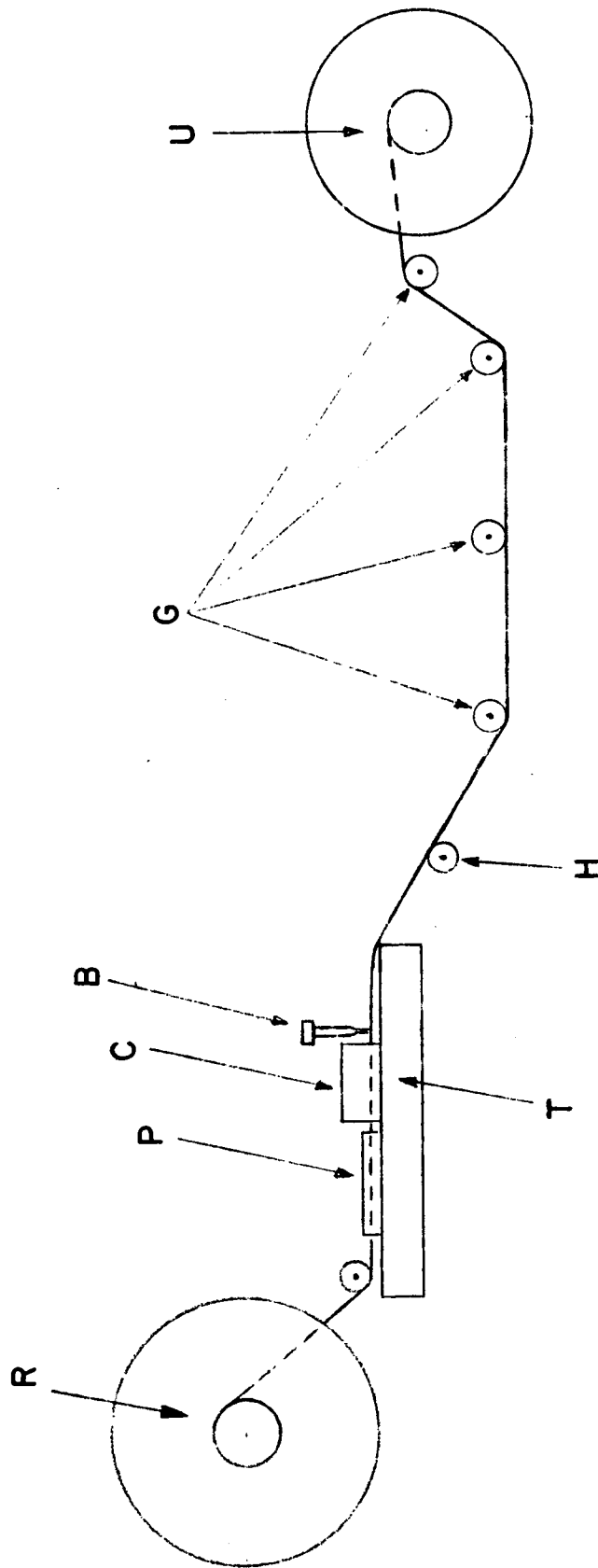


Fig. 2 TAPE COATING APPARATUS  
(Schematic View)

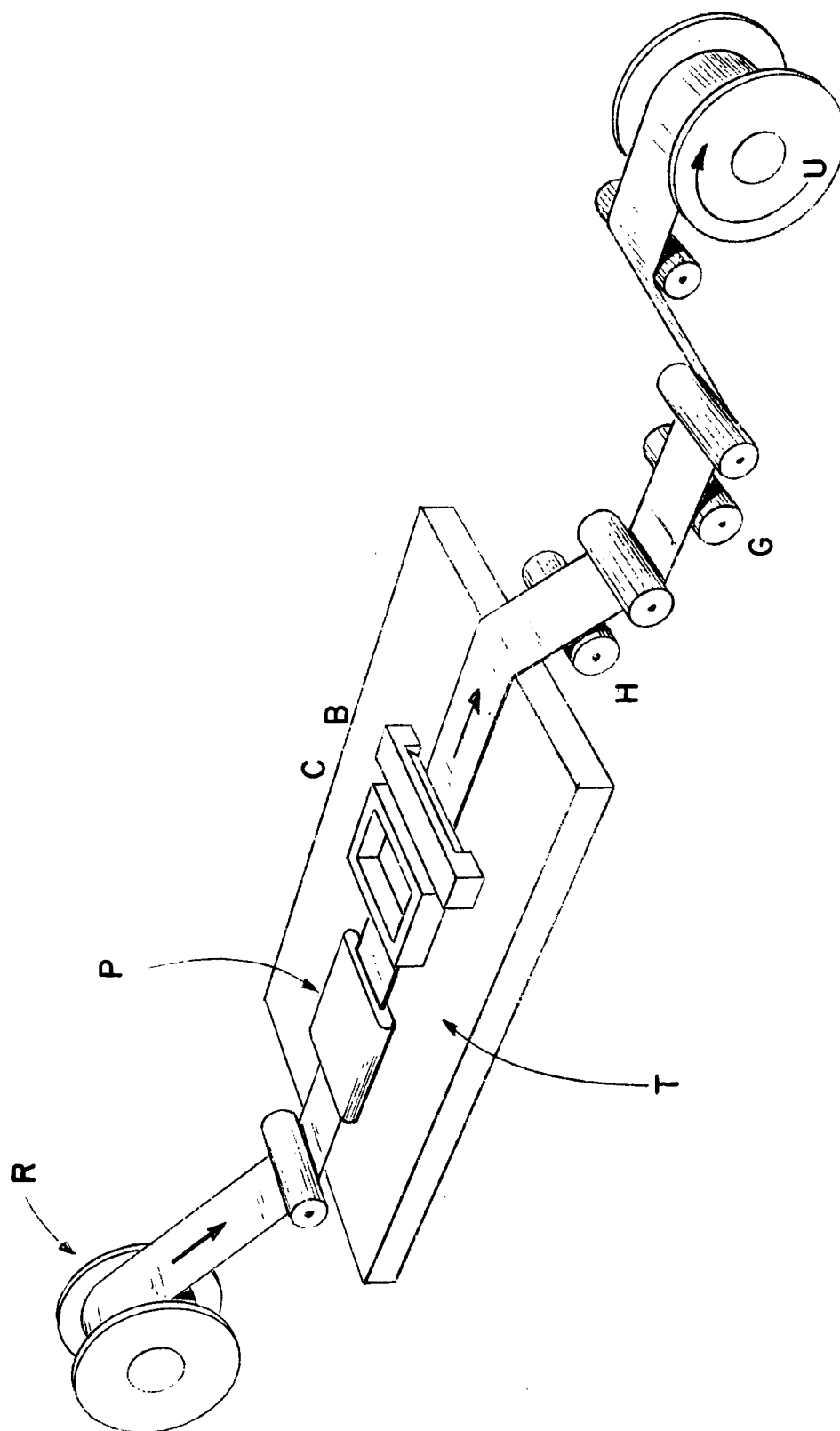


Fig. 3 TAPE COATING APPARATUS  
(General View)

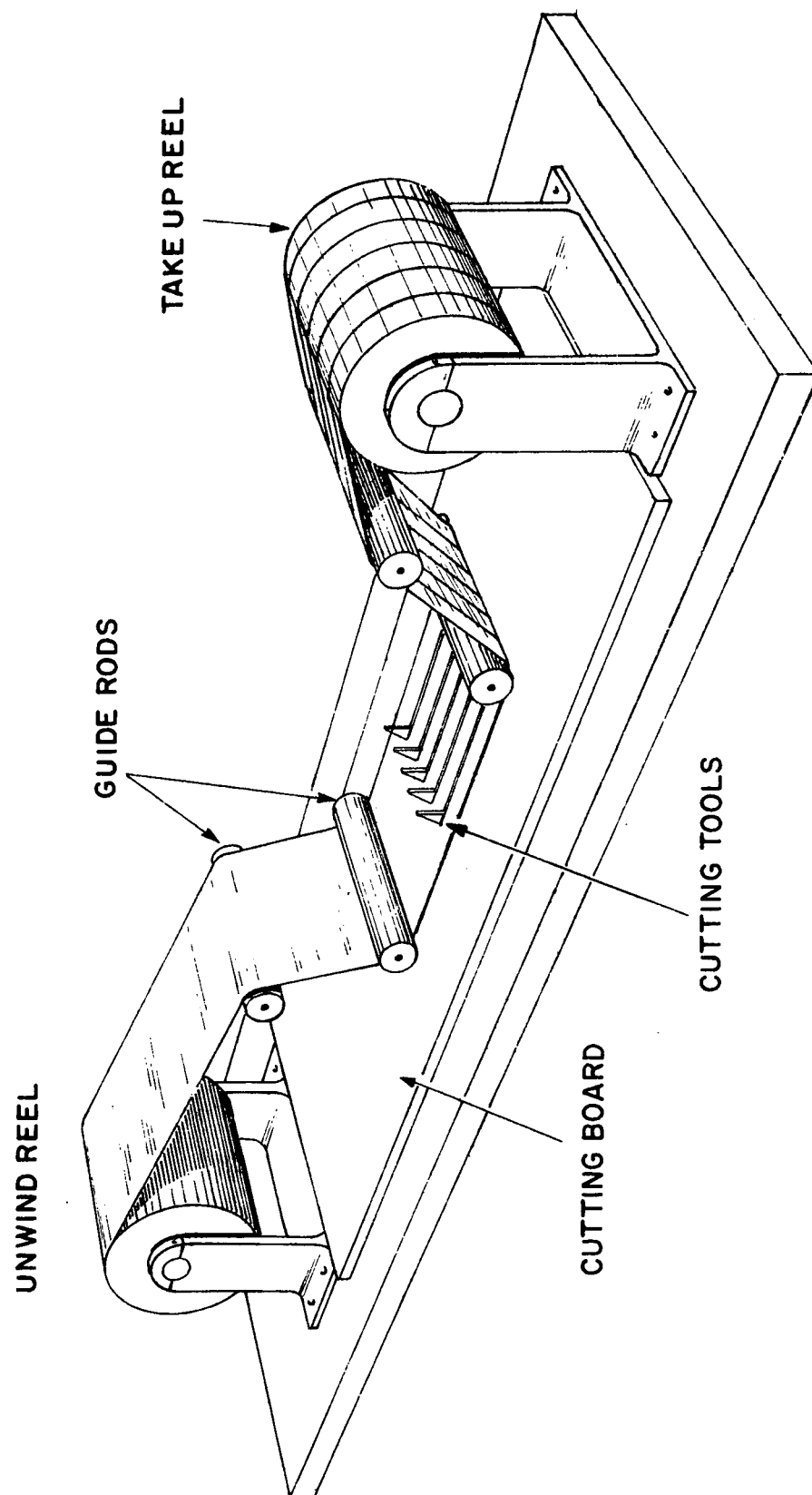


Fig. 4 TAPE SLITTER

phase consisted of 70% iron oxide pigment with 3% carbon black and 5% decafluorobiphenyl lubricant admixed in some formulations.

#### 4.3.5 Measurement of Structural Integrity of Coatings

The mechanical properties of unsupported films of magnetic coatings and unpigmented binder systems were intensively investigated in an effort to obtain information about the inherent strength of binders, their capability to accommodate magnetic oxides at high pigment levels, and the influence of additives on the structural integrity of the coatings. The measurements performed on an Instron testing machine involved the determination of stress-strain properties of unsupported, cured films of magnetic coatings (and unpigmented binder), that were cut into 4 x 0.5 x 0.003 in. specimens. These specimens, inserted into the jaws of the testing machine at 2 in. separation, were subjected to a progressively increasing tensile stress at a cross-head speed of 0.1 in./min. To expedite the evaluation of measured properties, the data, obtained in graphical form, were reduced by a digitizer whose output was processed by a programmable calculator-printer (Hewlett-Packard Co.). The thus obtained information included ultimate strength, ultimate elongation, modulus of elasticity and toughness. The latter, representing the integrated area under the stress-strain curve, was expressed in terms of the work expended to fracture a volume element of the film.

#### 4.3.6 Friction Measurements

The importance of the sliding behavior of instrumentation tape in contact with head surfaces of satellite recorders suggested the desirability of determining the frictional properties of developed tapes. The apparatus designed for this purpose (Figure 5) utilizes a modified ASTM D1894 test method for the measurement of the coefficient of friction of plastic materials. The test device consists of two polished glass guides, G, which are adhesively mounted on a 1/4-in. glass support plate, P, in such a way that the guides are separated from each other by a trough of 1/2-in width. A 1-1/2" x 1/2" x 1/2" steel sled, S, with Teflon, T, film bonded to the 1-1/2" x 1/2" face of the slider

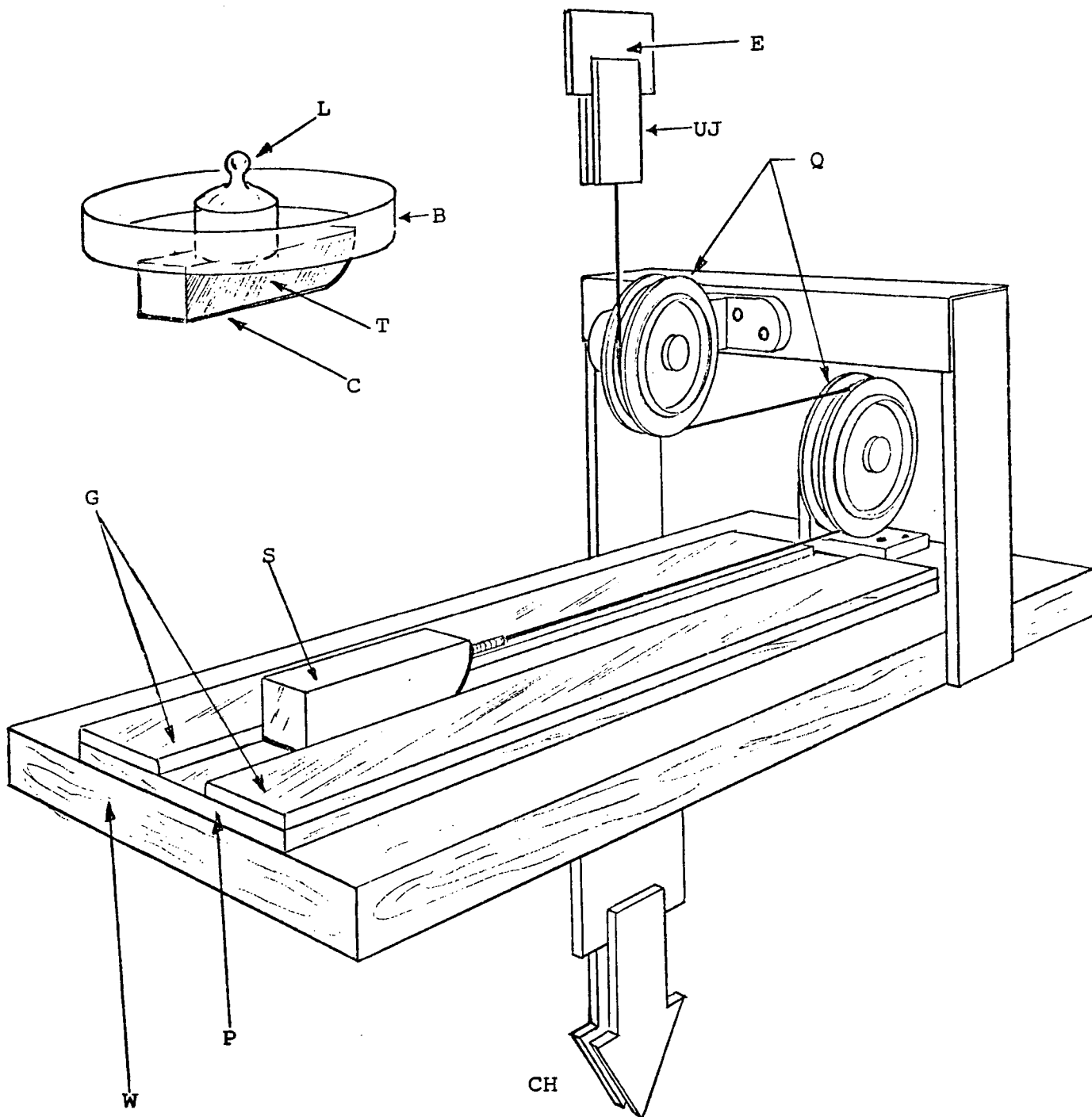


Fig. 5 TAPE FRICTION TEST APPARATUS

to minimize friction, comprises the moving element of the device. A bakelite pan, B, glued to the sled by means of epoxy resin facilitates placement of loads, L, on the sled. Copper shim stock, C, of 5-mil gauge is mounted on the contact surface of the sled by means of double-faced adhesive tape, to permit measurement of the coefficient of friction between a copper reference surface and the tape coatings studied. The support for the friction apparatus is provided by a 3/4-in. wood frame, W, which is affixed to the cross-head, CH, of an Instron testing machine. A pulley, Q, converts the vertical movement of the cross-head to a tangential movement of the sled. The sled is connected through the pulley and the upper jaws, UJ, of the Instron testing machine to a force-sensing cell, E. Thus the frictional force is measured while the sled moves tangentially on the coating surface. The tape is fixed in the trough between the glass guides and remains in contact with the sled at a sliding speed of 1 in/min and a normal load that can be varied by the use of appropriate weights.

The resistance to sliding offered by different tape coatings was continuously recorded during the traverse of the rider. The coefficient of friction could easily be computed from the measured frictional force and the load applied to the slider. A small computer program was also written to permit direct determination of the coefficient of friction and standard deviation for data in graphical form.

#### 4.3.7 Measurement of Thermal Properties

In an effort to obtain information about the temperature sensitivity and susceptibility of different magnetic coatings to adhere to a metal substrate at elevated temperatures, several thermal stability tests have been conducted on tape specimens. Initial tests were performed on a Kofler thermal gradient stage, in which 1/2-in.-wide tape strips were placed face-down on an aluminum foil-lined heating bar that maintained a temperature differential of 150°C (50 to 200°C). Contact between the magnetic coating and heated aluminum foil was established by placing a glass bar on the tape. Apparently the compressive load exerted by this bar was insufficient to insure intimate contact between the mating

surfaces, as the tape showed no tendency to adhere to the substrate in the entire thermal range of the heating stage.

To alleviate the problem of apparent metal/coating separation, an alternative test method was employed. In this method, magnetic coatings were applied to 1/2 x 4 in. tin foil or Mylar strips. These were placed face down on acid and solvent treated 0.003 in gage copper or 0.1 gage aluminum sheet such that only 1 or 2 in lengths of the coated strip contacted the metal substrate while the remainder provided a tab like appendage. A total of 4 to 8 test specimens were simultaneously introduced into a press whose heated platens exerted a pressure of 4000 psig, or 500 to 1000 psi per individual specimen, depending on their number and effective contact area. The temperature of the platens, controlled within 2°C, was maintained at 150, 175 and 200°C while the samples were kept in the press for five min. After exposure, the samples were removed from the press, cooled to 25°C, and inspected for adhesion by peeling off the tab from the metal substrate. This qualitative test produced useful results, inasmuch as it permitted differentiation between coatings obtained from different binders and also provided information about the effects of pigment content on the susceptibility of coatings to adhesion.

More quantitative measurements of the thermal stability of candidate magnetic tape coatings were made by thermogravimetric (TGA) methods in air and nitrogen atmospheres. In an effort to use comparable exposure conditions in both experiments, the air and nitrogen gas flows were maintained constant at an admission rate of 150 ml/min. The samples employed comprised 100 mg of fully cured coating material that was mechanically detached from Mylar or Kapton substrates. The samples were heated at a rate of 10°C/min while their weight was continually recorded on a Cahn electrobalance.

#### 4.4 Experimental Results and Discussion

##### 4.4.1 Mechanical Properties of Binder Systems and Coatings

The mechanical properties of the different binders considered in this study, as well as magnetic coatings prepared from them have been subjected to evaluation. Marked differences in structural integrity and capability of pigment accommodation have been observed for the binders already in the casting and curing processes. Thus, a polyester-based urethane (E-320/E-331) exhibited little binding capability for iron oxide and was therefore eliminated from further consideration. A modified polysiloxane resin (6103-60) with pronounced elongation characteristics and low strength could not be used by itself as a binder, but was employed as a component in inherently rigid polyurethane (X-4510-40) and polyepoxide (DER-669) resin systems. The mechanical behavior of these systems is discussed in the following sections.

##### 4.4.1.1 Epoxide Coatings

The brittleness of 0.003 in.-thick films of magnetic coatings prepared from 100% aromatic epoxide resin (DER-669) suggested modification of this binder by incorporating in it a flexibilizing polyamide (Versamid-115) co-reactant. The effects of this co-reactant on the strength of the binder system and its pigment-accommodating capability were determined for compositions ranging in epoxy:amide content from 75:25 to 30:70. The mechanical properties of clear films and those containing iron oxide pigment at different loading levels are summarized in Table 2.

As indicated, the addition of polyamide appears beneficial to the strength and toughness of the resin at lower concentrations of the additive. The addition of iron oxide pigment to the 75:25 epoxide:amide resin, which exhibited good mechanical properties in the series considered, lowered the strength of composite films in comparison with the unpigmented material. However, even at the 70% pigment-loading level, the coating maintained a strength and toughness of 794 and 1.06 psi, respectively. These values are comparable with those obtained at the same pigment content for the highly flexibilized 30:70 epoxy:polyamide system. It is



Table 2

## MECHANICAL PROPERTIES OF MODIFIED EPOXIDE BINDER AND MAGNETIC COATING

Sample Identifi- cation	Coating Composition, % by Weight of Total Resin			Mechanical Properties			
	<u>Epoxide</u>	<u>Polyamide</u>	<u>Pigment</u>	<u>Ultimate Strength, psi</u>	<u>Elonga- tion, %</u>	<u>Modulus psi x 10<sup>-6</sup></u>	<u>Toughness in-lb/in<sup>3</sup></u>
9	30	70	0	721	0.63	0.14	2.22
10	50	50	0	1,534	1.05	0.16	8.06
11	75	25	0	5,541	4.0	0.20	124
13	30	70	33.5	1,942	3.3	0.11	40.0
20	30	70	50	718	0.33	0.27	1.22
19	30	70	70	642	0.40	0.17	1.04
14	50	50	33.5	1,940	1.3	0.17	11.3
15	75	25	33.5	2,774	1.4	0.22	20.2
18	75	25	50	1,773	0.23	0.82	6.3
17	75	25	70	794	0.33	0.20	1.06

apparent that the cohesive strength of the coatings is not improved by the use of amide flexibilizer in excessive amounts. These considerations and the desired maintenance of a high pigment/binder ratio suggested the use of coating No. 17 for additional investigation of composition-related effects.

In a parallel effort to reduce the brittleness of aromatic epoxide coatings, DER-669 was reacted with 5 and 10% siloxane resin (6103-60). The effects of the co-reactant and an auxiliary curing agent (methylene dianiline) on the structural integrity of the coating in relation to the amount of pigment used were evaluated. The results shown in Table 3 confirm the previously observed reduction of the ultimate strength and elongation of epoxide-based coatings with increasing pigment level. The 20% retention of the ultimate strength of coatings that contained 70% iron oxide in comparison with those that had a 50% pigment level suggested a considerable increase in flaw centers as a result of the failure of the resin to accommodate greater amounts of pigment. It is conceivable that this accommodation could be improved by pretreatment of iron oxide or the use of additives in the resin. These approaches have not been applied in order to more effectively evaluate the compatibility of the binder proper with pigment.

It is interesting to note that, although the silicone resin reduced appreciably the strength of coatings on transition from a 50 to 70% pigment level, a 5% change in silicone content at constant (70%) pigment level had little effect on mechanical properties of coatings. Therefore, the polysiloxane appears to exert an influence not so much on the mechanical properties of the binder, as on its affinity for iron oxide pigment. The reduction in ultimate strength of siloxane-modified polyepoxide coatings at the 50% pigment level from 7500 to 1100 psi on introduction of methylene dianiline curing agent suggests preferential reaction of this agent with the aromatic epoxide to the detriment of the siloxane-epoxide reaction. The presence of partly unreacted siloxane could be expected to introduce weak spots in the coating thereby reducing its strength. In view of these considerations, auxiliary curing agents should be omitted from silicone-modified epoxy binders.

Table 3  
MECHANICAL PROPERTIES OF SILICONE RESIN-MODIFIED EPOXIDE COATINGS

Sample Identification	% Weight of Coating Pigment	Coating Composition			Mechanical Properties		
		% by Weight of Total Resins	% by Weight of Epoxide		Ultimate Strength, psi	Elongation %	Modulus psi x 10 <sup>-5</sup>
			Epoxide	Auxiliary Curing Agent			
SEQ 17/2-4	70	95	5	0	1,870	1.6	0.17
SEQ 17/5-8	70	90	10	0	2,200	1.2	0.18
SEQ 17/9	70	95	5	25	654	0.3	0.24
SEQ 17/11-14	70	90	10	25	513	0.3	0.23
SEQ 19/49-51	50	90	10	0	7,470	2.8	0.42
SEQ 19/56, 57	50	90	10	25	1,120	0.3	0.38
							18.8
							13.8
							1.1
							0.7
							130.0
							2.0

In an effort to determine the effect of structural variables of epoxide resins on the properties of coatings, an aliphatic polyepoxide (DER-736) was used as a counterpart of its aromatic (DER-669) analog. Films obtained from this resin were characterized by a relatively low strength (230 psi), low modulus ( $4.0 \times 10^2$  psi) and considerable elongation (50%). The resin exhibited pigment-binding capability at low (25%) iron oxide loading levels as indicated by the improved strength of the coating (1050 psi) in comparison with the unpigmented film (230 psi). However, the aliphatic resin, unlike its aromatic counterpart, could not accommodate 70% pigment. For this reason, and in view of the later discussed friction relationships, the aliphatic epoxide was discounted as a suitable binder for instrumentation tape magnetic coatings.

Investigations of the influence of carbon black and lubricating agents on the mechanical properties of a polyamide-modified aromatic epoxide coating (Sample 17, Table 2) disclosed apparent beneficial effects of carbon loading on the mechanical properties of magnetic coatings at the 3% carbon level (Table 4). However, at 5% concentration of the conductivity-improving additive, the coating remained no longer coherent. The reinforcing action of carbon on the strength of composite films is thus limited to lower carbon contents of iron-oxide-loaded coatings. The fluorinated lubricants, selected because of their potential friction-reducing capability, affected the mechanical properties of the coating to a different extent depending on the nature of the fluorinated compound. Thus heptafluorobutyramide, which had little effect on the mechanical properties of the coating when used in an amount of 2%, reduced the cohesive strength of the coating appreciably when employed at higher lubricant concentrations. By contrast, decafluorobiphenyl had a much lesser effect on the mechanical properties at the three concentration levels tested. Investigations involving a perfluoro-alkane lubricating additive, disclosed complete loss of structural integrity of the magnetic coating after addition of 2% of this compound to the coating system. Thus, the importance of the nature and amount of

Table 4  
EFFECTS OF ADDITIVES ON MECHANICAL PROPERTIES OF EPOXIDE COATING SYSTEM\*

Sample Identifi- cation	Additive		Mechanical Properties			
	Nature	Quantity % by weight of resin	Ultimate Strength, psi	Elongation %	Modulus psi x 10 <sup>-6</sup>	Toughness, in-lb/in <sup>3</sup>
17	None	0	794	0.33	0.20	1.06
33	Carbon black	3	994	1.3	0.2	8.75
34	Carbon black	5	Film too weak to test			
35	Lubricant: Heptafluorobutyramide	2	797	0.6	0.2	2.43
36	Heptafluorobutyramide	5	436	0.3	0.14	0.38
37	Heptafluorobutyramide	8	105	0.2	0.05	0.10
38	Decafluorobiphenyl	2	516	0.42	0.16	1.02
39	Decafluorobiphenyl	5	512	0.40	0.18	0.85
40	Decafluorobiphenyl	8	551	0.41	0.15	1.20

\*Coating No. 17: Binder: Epoxy/polyamide (75% DER-669, 25% Versamid 115)  
Pigment: Gamma iron oxide (70% by weight of resin)

lubricating additives with regard to the integrity of magnetic coatings is clearly emphasized.

#### 4.4.1.2 Polysiloxane-Urethane Coatings

The pigment-loading capability and mechanical strength of polyester-modified silicone/urethane (6103-60/X-4510-40) coatings have been investigated by stress-strain measurements in accordance with the previously used procedure. The data, summarized in Table 5, indicate the low strength, low modulus and pronounced elongation of the unpigmented silicone polymer. However, when this silicone was used as a co-reactant with an inherently rigid urethane resin in a proportion of 57/43 silicone/urethane it imparted good mechanical properties to the unpigmented binder system (5000 psi, 6% elongation). Nevertheless it was disappointing to find that the strength of coatings prepared from the binder at 50% pigment content was greatly diminished in comparison with the unfilled system. This could be attributed to the poor pigment binding capability of the silicone polymer, since coatings that contained the same amount of iron oxide, but a lesser (25%) proportion of silicone, exhibited better mechanical properties. The deficient thermal properties of the siloxane-urethane binder and the difficulty of obtaining coherent coatings from formulations that contained more than 50% iron oxide made it inadvisable to further consider this binder for coating applications.

#### 4.4.1.3 Polyamide Coatings

In an effort to evaluate the performance of a flexible, pigment-receptive binder, in comparison with the rigid epoxide binder previously investigated, attention has been given to the mechanical properties of coatings prepared from a polyamide (Elvamide 8061) resin, at pigment contents ranging from 25 to 70%. The results, summarized in Table 6, indicate a relatively low strength of unpigmented films. However, an interesting feature of the mechanical behavior of iron oxide-filled polyamide films is the relatively constant strength over a wide range of pigment content. This might suggest cohesive failure of the resin between well enveloped pigment particles, or cancelation

Table 5

## MECHANICAL PROPERTIES OF SILICONE-URETHANE-BASED MAGNETIC COATINGS

Sample Identifi- cation	Coating Composition		Curing Conditions	Mechanical Properties			
	Resin System	Pigment Content, % by Wt. of Resin		Ultimate Strength, psi	Elonga- tion, %	Modulus- psi x 10 <sup>-6</sup>	Toughness in-lb/in <sup>3</sup>
I-11	6103 (100*)	0	1 wk at 25°C	630	79	0.03	361
I-12	6103 (100)	0	1 wk at 25°C 2 min at 200°C	780	30	0.02	207
I-13	6103/X-4510 (57/43)	0	1 wk at 25°C	4,700	5.2	0.17	152
I-14	6103/X-4510 (57/43)	0	1 wk at 25°C 2 min at 200°C	5,200	6.7	0.19	244
26	6103/X-4510 (53/47)	0	2 hr at 75°C 1/2 hr at 175°C	5,369	4.6	0.20	134
27	6103/X-4510 (53/47)	0	2 hr at 75°C 1/2 hr at 175°C	4,950	4.5	0.20	123
SEQ-7	6103/X-4510 (40/60)	25	2 hr at 75°C 1/2 hr at 175°C	440	46	0.02	162
SEQ-10	6103/X-4510 (40/60)	50	2 hr at 75°C 1/2 hr at 175°C	220	85	0.006	138
SEQ-11	6103/X-4510 (25/75)	50	2 hr at 75°C 1/2 hr at 175°C	960	34	0.08	280

Table 6

MECHANICAL PROPERTIES OF POLYAMIDE COATINGS AT 25%, 50%, AND 70% PIGMENT CONTENT

<u>Sample Identification</u>	<u>Coating Composition % by Weight</u>		<u>Mechanical Properties</u>			
	<u>Resin</u>	<u>Pigment</u>	<u>Ultimate Strength psi</u>	<u>Elongation %</u>	<u>Modulus-6 psi x 10</u>	<u>Toughness in-lb/in<sup>3</sup></u>
SEQ 20/17-20	100	0	3,100	106	0.05	2,310
SEQ 20/21-24	75	25	3,030	12.9	0.09	297
SEQ 20/25-28	50	50	3,040	11.0	0.12	263
SEQ 20/29-32	30	70	2,460	1.7	0.21	26.3



of the reinforcing effect of pigment by its introduction of weak spots in the coating.

#### 4.4.1.4 Polycarbonate-Amide Coatings

In evaluating the pigment-binder compatibility and mechanical properties of an experimental carbonate-amide polymer that was synthesized at IITRI, clear and pigmented films were prepared from an AC-2 resin which had an intrinsic viscosity of 0.68. The coating integrity, strength, modulus and toughness of these coatings were good, as shown in Table 7.

Attempts to correlate the properties of the coating with molecular weight were unsuccessful, as the solution viscosity of the AC-2 resin could not be exceeded in subsequently synthesized products, and the polymer fractions of lower inherent viscosity produced fragile films. It has been observed that the synthesis of carbonate-amide polymers in a molecular weight range that would permit formulation of coatings depended to a critical extent on the admission rate of phosgene to the reaction system and on the purity of the isophthaloyl chloride reagent. Problems encountered in reproducing the properties of synthesized resins have been alleviated by close control of reaction conditions and purification (re-crystallization) of reagent compounds.

#### 4.4.1.5 Polyimide-Based Coatings

The use of a binder with exceptional thermal stability and good mechanical properties for the development of durable instrumentation tape coatings was investigated in coatings prepared from a polyimide (DE 910-101) resin. The test data presented in Table 8 indicate that the polyimide resin exhibits very good mechanical characteristics. However, these characteristics tend to deteriorate with the introduction of increasing amounts of iron oxide, as indicated by the reduction of the ultimate strength and elongation from 7,300 psi and 2.1% to 2,800 psi and 0.9% for film specimens that contain 25 and 70% pigment, respectively. In absolute terms, the 70/30 pigment/resin composition of polyimide coatings shows a noticeable improvement in mechanical properties over silicone-modified epoxy coatings of same pigment content.

MECHANICAL PROPERTIES OF CARBONATE-AMIDE AC-2 BINDER AND PIGMENTED COATINGS

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Table 8

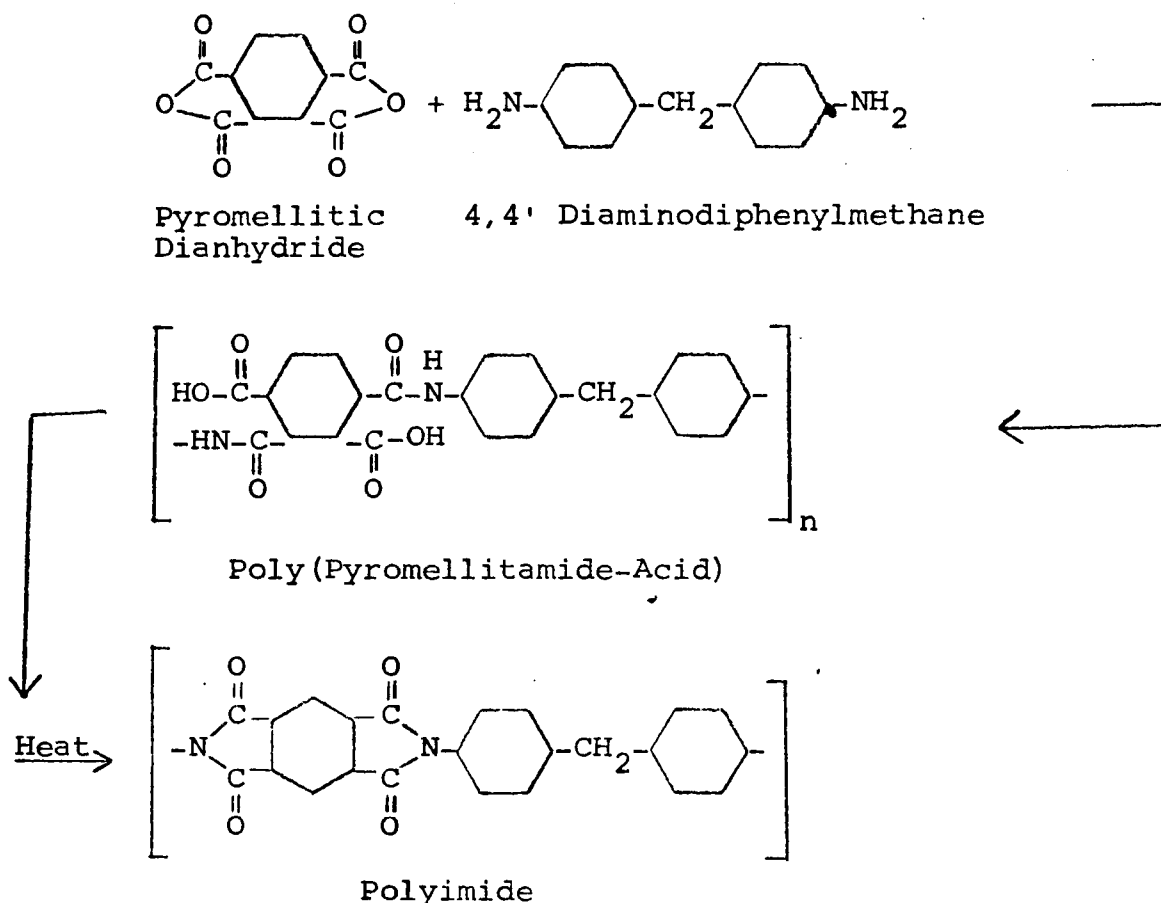
MECHANICAL PROPERTIES OF POLYIMIDE COATINGS AT 25%, 50%, AND 70% PIGMENT CONTENT

<u>Sample Identification</u>	<u>Coating Composition % by Weight</u>		<u>Mechanical Properties</u>		
	<u>Resin</u>	<u>Pigment</u>	<u>Ultimate Strength, psi</u>	<u>Elongation %</u>	<u>Modulus psi x 10<sup>-6</sup></u>
SEQ 19/33-36	100	0	14,800	9.3	0.33
SEQ 19/37-40	75	25	7,290	2.1	0.40
SEQ 19/41, 42	50	50	4,600	1.1	0.46
SEQ 21/64-66	30	70	2,800	0.9	0.46
					975
					64
					26
					15.4

It should be pointed out that the strength and elongation properties of polyimide coatings could conceivably be improved by a better envelopment of individual pigment particles in the resin matrix. Such envelopment would be promoted by extending the milling time, use of surface treatments for the pigment and introduction of dispersion-promoting agents into the coating system. Also, as the toughness of cured films has been found to depend on the completeness of the imide cyclization process, better mechanical properties could be obtained from coatings that have received an optimum cure. In this regard, an attempt has been made to ascertain the degree of imide cyclization by infrared (I.R.) analysis of unpigmented films that were subjected to different curing cycles.

The full curing cycle involved a heating period of 50 min at 75°C, followed by exposure to 150°C for 2 hr and 260°C for 15 min. The corresponding I.R. spectra for the three consecutive "curing" stages are shown in Figure 6 (A through C).

In interpreting these spectra it must be realized that in most applications of "polyimide" resins from solution, the polymer is more likely represented by a poly(amide-acid) which only through heat treatment (or chemical reaction) can be converted to a polyimide with desired heat stability and mechanical properties. The preparation of the polyimide can be represented by the following example:



It is apparent that, in the conversion of the poly(amide-acid) to polyimide, the Amide I and II bands representing  $\nu(\text{C}=\text{O})$  and  $\delta(\text{NHR})$  stretching and bending vibrations at 1670 and 1540  $\text{cm}^{-1}$ , respectively, should disappear, while new bands, characteristic of the cyclic amide should gain prominence. The latter absorption bands occur at 1775 and 1317  $\text{cm}^{-1}$ , as indicated in Figure 6. The spectroscopic changes that occurred in the final cure stage at 260°C, specifically the disappearance of the 1540  $\text{cm}^{-1}$  band and the considerable peak reduction at 1670  $\text{cm}^{-1}$ , indicate gross conversion of the resin to its cyclized imide structure. It would remain to be shown whether this conversion could still be furthered by prolonging the time of final cure or by raising the temperature of the final thermal treatment. Any benefits derived from this treatment in regard to mechanical properties of the coating would then have to be ascertained.

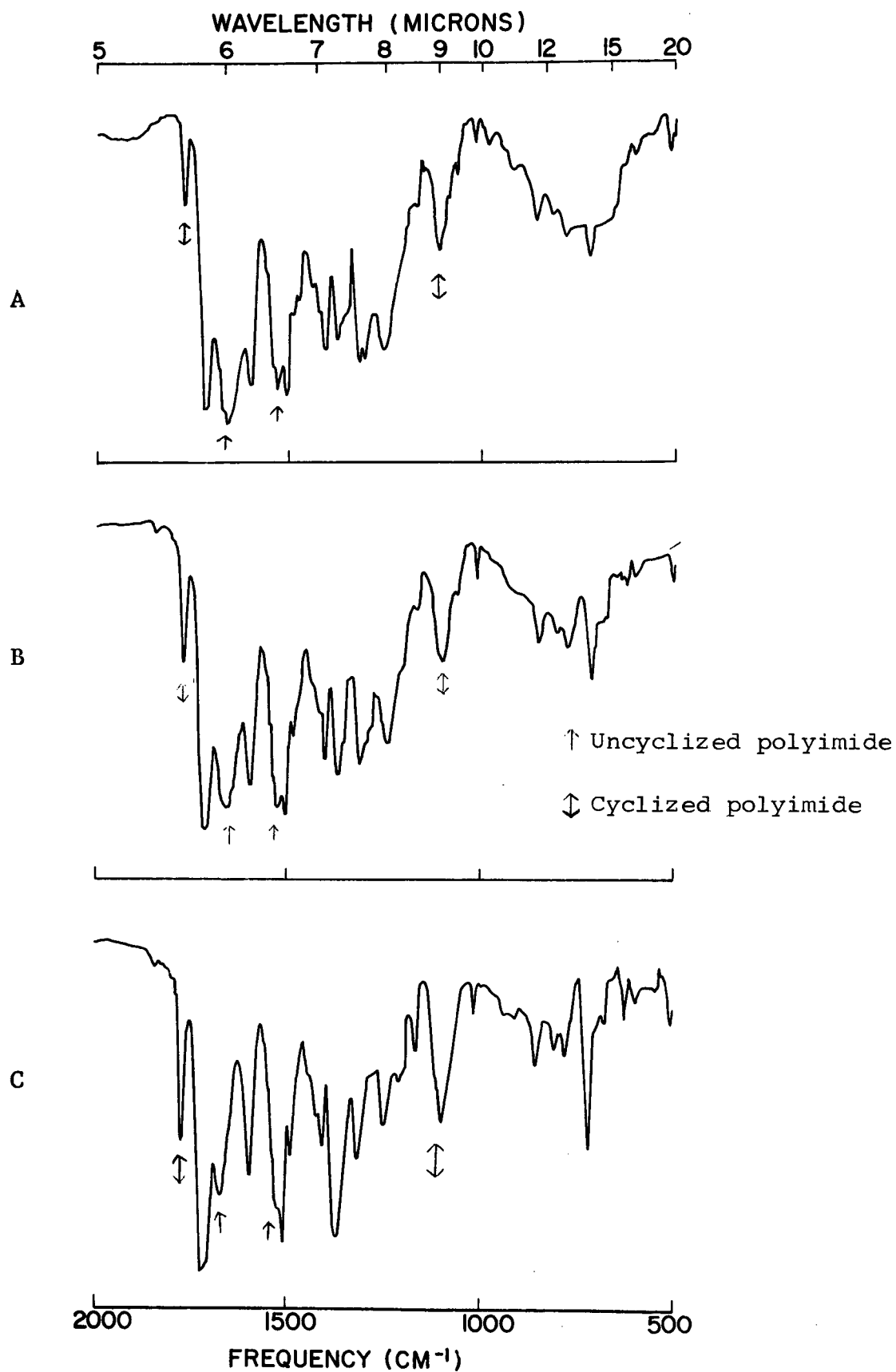


Figure 6  
INFRARED ABSORPTION BANDS OF POLYIMIDE COATINGS

#### 4.4.2 Frictional Behavior of Selected Coatings

In an effort to determine the surface properties of several magnetic coatings with regard to their friction behavior, the coefficients of friction of aromatic (DER-669) and aliphatic (DER-736) epoxide coatings as well as of coatings obtained from the experimental amide-carbonate polymer were determined, with particular consideration of the effect of pigment level on the sliding properties of tape. For comparison, the coefficients of friction of commercial instrumentation tapes (3M 888 and RCA 617) have also been determined. The normal load in these measurements was maintained at 175g, and the sliding speed was 1 in/min.

The data, summarized in Table 9, show a substantially higher coefficient of friction of aliphatic epoxides in comparison with aromatic epoxy compounds at equivalent pigment loadings. The high standard deviation ( $\pm 0.29$ ) of the coefficient of friction of the aliphatic epoxide coating was due to the stick-slip movement of the sled on this coating. Apparently, the greater chain flexibility of aliphatic resins is responsible for intermittent locking of chain segments at the active sites of the metal surface. The preference of more rigid (aromatic) polymer binders may thus be suggested on the basis of head/tape friction considerations.

The frictional behavior of clear and iron oxide-filled carbonate-amide coatings suggests a lessened frictional resistance to sliding of coatings that contain a greater amount of pigment. This might be attributed to the lower effective contact area between the sliding surfaces for coatings that contain a greater proportion of pigment and the lessened binder/metal interaction as a result of the diminished availability of functional groups on the binder for interaction with the substrate, because of prior blocking of these groups by pigment-binder interaction. Thus, as long as the binder is capable of accommodating larger amounts of magnetic oxide, a high pigment loading level might not have adverse effects on the transport of tape across the recording head surface. This, of course, assumes maintenance of good surface finish properties for the coating and minimization of oxide

Table 9

## FRICTION PROPERTIES OF MAGNETIC COATINGS AND TAPES

Specimen Sequence No.	Sample Identification		Coefficient of Friction Cu/Coating	Standard Deviation
	Resin	Iron Oxide by Weight of Resin, %		
133-137	Aliphatic epoxide (DER-736)	25	0.52	0.29
111-115	Aeromatic epoxide (DER-669)	25	0.17	0.02
51-55	Carbonate-amide (AC-20)	0	0.24	0.01
81-85	Carbonate-amide (AC-20)	50	0.20	0.02
96-100	Carbonate-amide (AC-20)	70	0.14	0.01
37-40	Polyester-urethane (3M 888)	@75	0.21	0.02
6-10	Polyester-urethane (RCA 617)	@70	0.25	0.03



shedding problems. An attempt to upgrade the frictional characteristics of the aliphatic epoxy resin in magnetic coatings by increasing the pigment loading level from 25 to 70% failed, as the binder could not retain that amount of pigment.

In comparing the coefficients of friction of the experimental coatings with those obtained for commercial instrumentation tape, the results, with the exception of the aliphatic epoxide coating, are not greatly disparate. Actually, the AC-20 coating containing 70% pigment has a lower coefficient of friction ( $0.14 \pm 0.01$ ) than commercial 3M 888 tape ( $0.21 \pm 0.03$ ).

#### 4.4.3 Thermo-Adhesive Properties of Coatings

The temperature sensitivity and susceptibility of different magnetic coatings to adhesive seizure were investigated for samples that were brought into contact with copper or aluminum substrates under a compressive force (500 to 1000 psi) at temperatures of 150 to 200°C, in accordance with the previously discussed procedure (Section 4.3.7). It has been observed (Table 10) that the presence of an aliphatic epoxide in epoxide binders appeared to enhance the susceptibility of the binder to adhesion. This behavior was also observed in preliminary tests on pigmented 50/50 DER-669/DER-736 coatings that contain 50% iron oxide. In parallel tests on a 75/25 aromatic epoxy/amide system, the tendency of pigmented coatings to adhere to copper substrates was considerably lessened. Here, coatings that contained 70 as well as 50% iron oxide exhibited no adhesive tendencies when compressed at 200°C, although under similar conditions, unpigmented and 25% iron oxide-containing coatings showed signs of transfer of adhesive to copper and aluminum substrates. Attempts to alleviate these effects by the use of fluorinated release agents, that were applied to the surface of cured coatings as a very thin film, proved unsuccessful. Although the observed failure of release agents could be attributed to the inappropriate choice of compounds employed, it would appear desirable to avoid secondary binder additives for minimizing adhesive interactions between magnetic coatings and head surfaces.

FOLDOUT FRAME 1

FOLDOUT FRAME 2

Table 10  
THERMO-ADHESIVE PROPERTIES OF MAGNETIC COATINGS

Coating System		Other Binder Additives	Tape Substrate	Contact Substrate	Temperature, °C	Observations About Coating in Peeling
Binder Composition	Iron Oxide Content, %					
<u>Epoxides</u>						
Aromatic/Aliphatic DER-669/DER-736						
50/50	0	0	Tin foil	Cu	200	Adhesion and transfer to Cu
75/25	0	0	Tin foil	Cu	200	Adhesion and transfer to Cu
85/15	0	0	Tin foil	Cu	200	Adhesion and transfer to Cu
<u>Epoxy-Amides</u>						
DER-669/Versamid 115						
75/25	0	2% DFBP <sup>a</sup>	Tin foil	Al	150	Very little adhesion
75/25	0	2% DFBD	Tin foil	Al	200	Pronounced adhesion
75/25	70	0	Mylar	Cu	200	No adhesion
75/25	70	5% DFBD	Mylar	Cu	200	No adhesion
75/25	70	SR-173 <sup>b</sup>	Mylar	Cu	200	No adhesion
75/25	50	0	Mylar	Cu	200	No adhesion
75/25	50	3% C	Mylar	Cu	200	No adhesion
75/25	50	5% DFBD	Mylar	Cu	200	No adhesion
75/25	50	SR-173, 2%	Mylar	Cu	200	No adhesion
75/25	25	0	Tin foil	Cu	200	Adhesion and transfer to Cu
75/25	25	MPO <sup>c</sup>	Tin foil	Cu	200	Adhesion and transfer to Cu
75/25	25	Teflon re- lease agent	Tin foil	Cu	200	Adhesion and transfer to Cu
<u>Carbonate-Amide</u>						
AC-20	0	0	Tin foil	Al	150	Slight Adhesion
AC-20	0	0	Tin foil	Al	175	Adhesion; no transfer to Al
AC-20	0	0	Tin foil	Al	200	Adhesion and transfer to Al
AC-20	25	0	Tin foil	Al	150	No adhesion
AC-20	50	0	Tin foil	Al	150	No adhesion
AC-20	70	0	Tin foil	Al	150	No adhesion
AC-20	25	0	Tin foil	Al	175	Slight Adhesion
AC-20	50	0	Tin foil	Al	175	No adhesion
AC-20	70	0	Tin foil	Al	175	No adhesion
AC-20	25	0	Tin foil	Al	200	Adhesion and transfer to Al
AC-20	50	0	Tin foil	Al	200	No adhesion
AC-20	70	0	Tin foil	Al	200	No adhesion
Elvamide-8061	50	0	Mylar	Cu	150	Pronounced Adhesion
Elvamide-8061	70	0	Mylar	Cu	150	Noticeable adhesion
DER-669 + 10% 6103 Silicone	50	0	Mylar	Cu	150	Slight adhesion
DER-669 + 10% 6103 Silicone	70	0	Mylar	Cu	150	No adhesion
Polyimide	50	0	Kapton	Cu	150	No adhesion
Polyimide	70	0	Kapton	Cu	150	No adhesion
Polyimide	50	0	Kapton	Cu	200	No adhesion
DER-669 + 10% Silicone	70	0	Mylar	Cu	200	Slight adhesion
Polyimide	70	0	Kapton	Cu	200	No adhesion

<sup>a</sup>Decafluorobiphenyl lubricating agent

<sup>b</sup>Reactive silicone lubricating agent

<sup>c</sup>Methyl perfluorooctanoate

The experimental carbonate-amide polymer exhibited thermo-adhesive characteristics similar to those of the aromatic epoxide-amide polymer. Coatings that contained more than 25% iron oxide had no adhesive tendencies when tested at 200°C. At a temperature of 150°C, even specimens that contained 25% pigment, exhibited no signs of adhesive interaction with aluminum substrates. These results correspond with the previously reported relationship between the coefficient of friction and the iron oxide level. In both cases, adhesive effects were minimized in coatings with a greater pigment/binder ratio. The cause underlying both effects might be the same, i.e., the diminished availability of functional groups in coatings with a greater proportion of pigment would minimize adhesion as well as reduce friction between sliding surfaces.

In an extension of the thermo-adhesive studies to polyamide, silicone-modified epoxide and polyimide coatings, the polyimide coatings were found to maintain their nonadhesive properties at low as well as high (70%) pigment levels (and at temperatures up to 200°C). By contrast, the polyamide and silicone-modified epoxide coatings exhibited a tendency to adhere to copper surfaces, when kept in compressive contact with these surfaces at 150°C for 5 min. The superior thermal properties of polyimide coatings are thus clearly emphasized.

#### 4.4.4 Thermogravimetric Measurements

The thermal stability of candidate magnetic tape coatings was investigated by thermogravimetric (TGA) methods in air and nitrogen gas atmospheres. Weight losses of these coatings, which contained 70% pigment in polycarbonate-amide (AC-26), polyamide-modified epoxide (DER 669/Versamid 115), polyamide (Elvamide 8061), and polyimide (DE910-101) binders, as well as of a commercial instrumentation tape coating, are shown in Figures 7 and 8. As indicated, the weight losses of the samples conditioned in air and nitrogen follow generally the same trend. The inert atmosphere represents a less damaging environment, particularly for the polyamide coating, although the magnitude of recorded weight losses is not strikingly different for both environments. The main differences

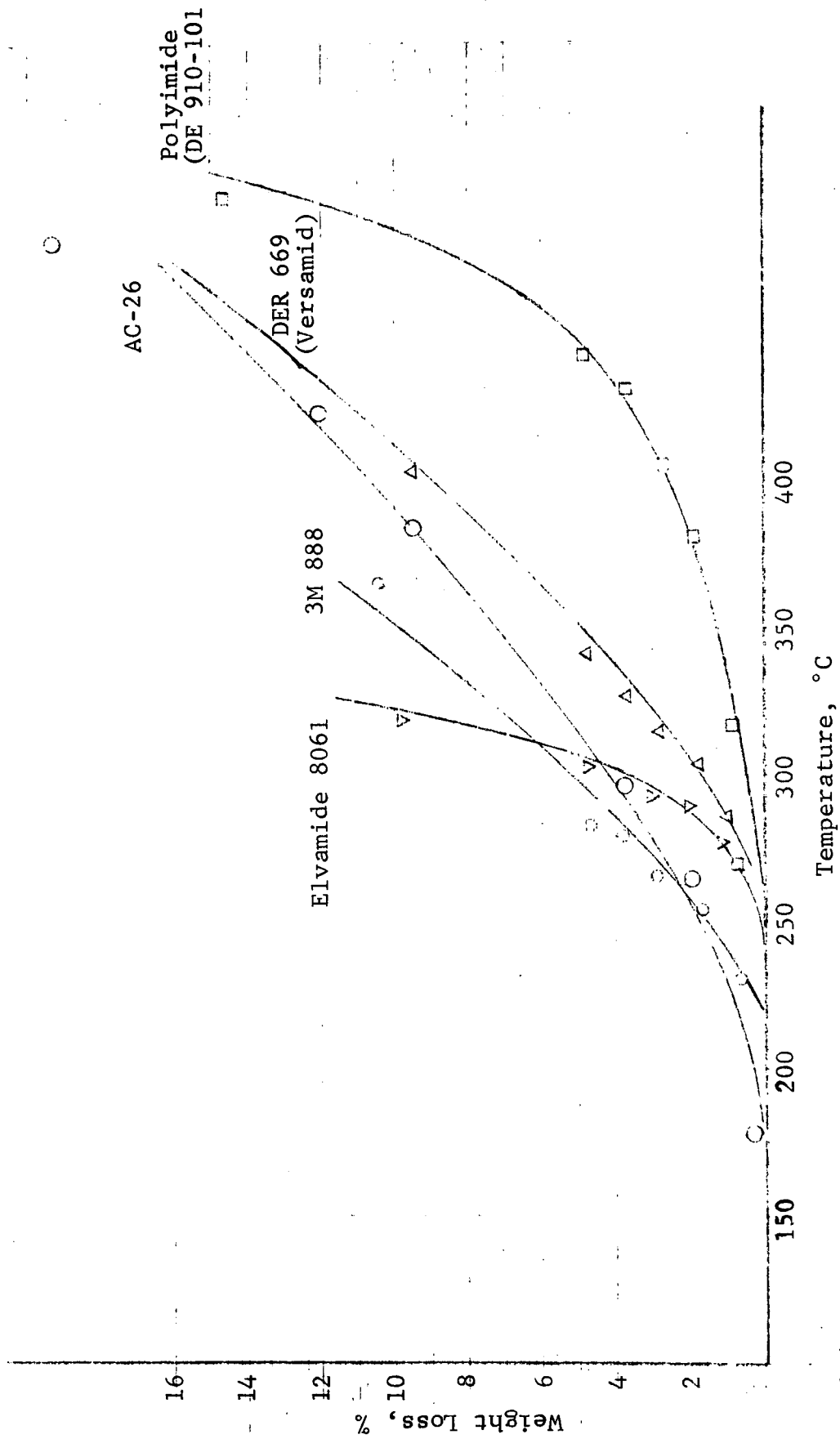


Figure 7  
WEIGHT LOSS OF MAGNETIC COATINGS IN AIR

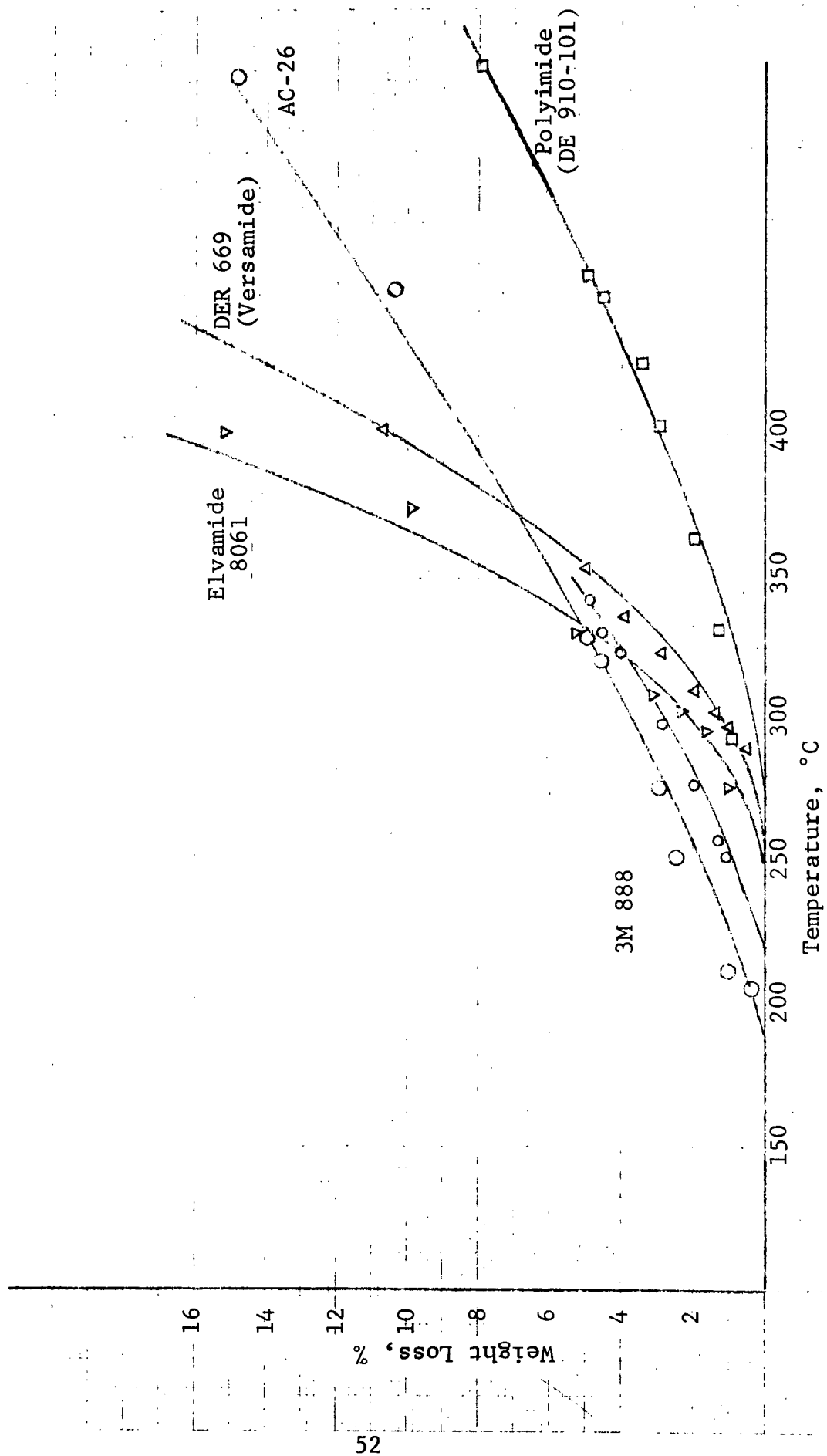


Figure 8  
WEIGHT LOSS OF MAGNETIC COATINGS IN NITROGEN

in behavior relate to the nature of the binder system. The polyimide is least affected by temperature, with the epoxide, polyimide, and carbonate-amide binders following in the order of increasing susceptibility to thermal effects. The carbonate-amide polymer appears to show a slight thermal instability in the initial heating range up to 300°C. However, above this temperature, the thermal behavior of the polymer shows improvement inasmuch as additional weight losses are smaller than those encountered for the polyamide and the commercial instrumentation tape coatings (3M-888). It is possible that the observed behavior of the carbonate-amide is related to the release of small amounts of entrapped solvent from AC-26 coatings.

TGA measurements, performed on polyimide films that were not fully cured, disclosed a significant weight loss at temperatures below 200°C, while the well-cured resin exhibited no perceptible weight losses almost up to 300°C (Figure 9). Since uncured polyimides eliminate water during cyclization, the observed loss of weight must be attributed to this cause. The importance of a complete cure of polyimide coatings is thus re-emphasized.

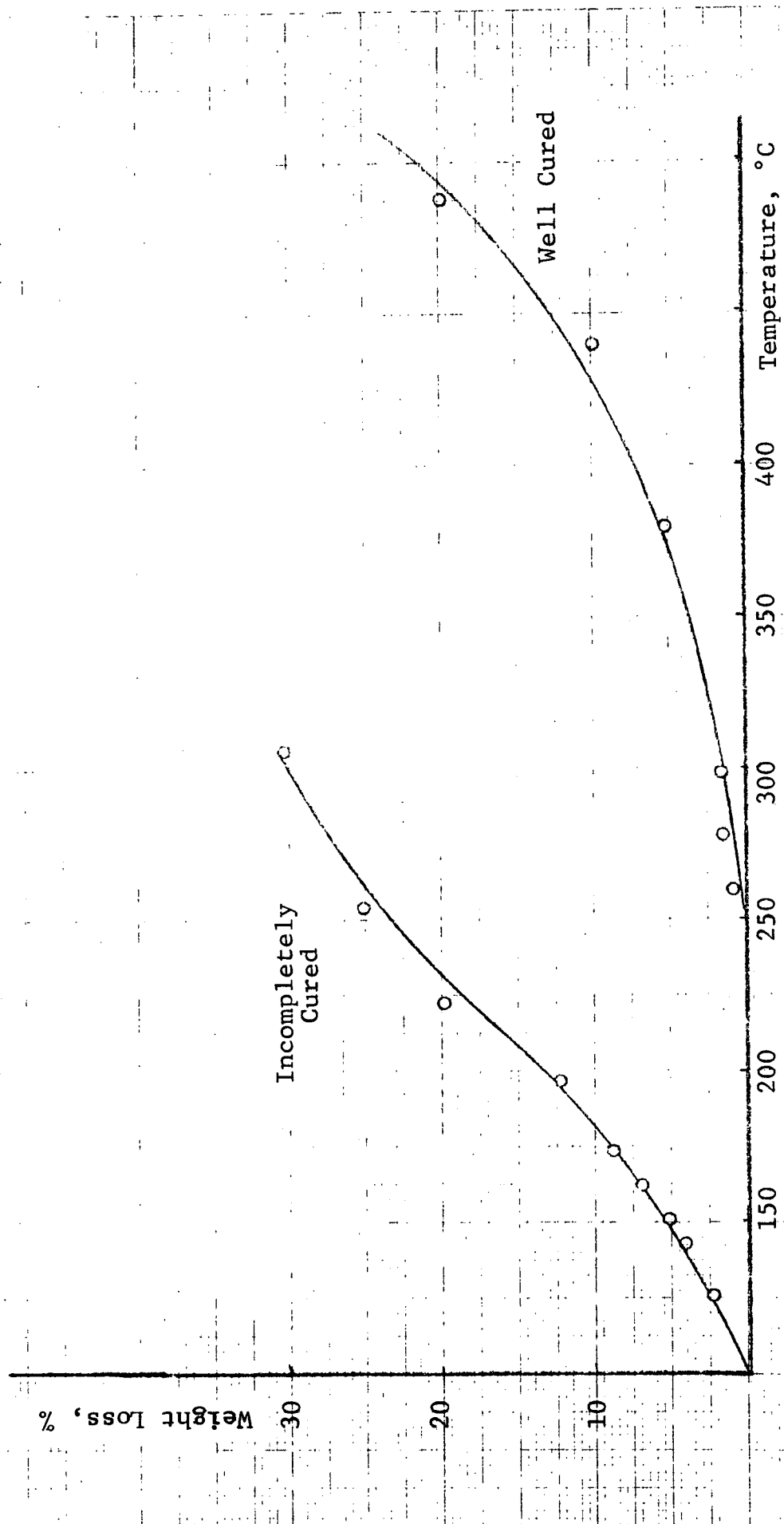


Figure 9  
WEIGHT LOSS OF INCOMPLETELY CURED AND WELL CURED POLYIMIDE COATINGS

## 5. PRELIMINARY ASSESSMENT OF TAPE PERFORMANCE

### 5.1 Short Tape Length Samples

Seven short length tape samples were made available for the assessment of their relative performance. These samples, ranging in length from between 12 to 25 feet and 1/2 inch wide, were prepared in the manner documented in Section 4.3.4 of this report. The seven tape samples consisted of three basic polymer types, an aromatic epoxide modified with a polyamide co-reactant (DER 669, Versamid 115), a polyamide (Elvamide 8061) and an experimental polycarbonate-amide (AC 26). Choice of the magnitude of the oxide loading together with the type and quantity of the other additives were arrived at from the results of experimental investigations described in Section 4. The seven tape samples evaluated during this preliminary tape assessment are described in more detail in Table 11.

### 5.2 Evaluation Tests

In order to fully evaluate the sample tapes the following tests were undertaken.

- Guidelines tests for tape selection established under Contract No. NAS5-11622. These included thermal stability, lubrication content, resistivity, chlorine content, oxide dispersion and flexibility.
- Coefficient of friction with various recording head materials including Aluminum, Havar, Alfesil, and combination of these in composite head structures.
- Magnetic properties of the tape samples including coercivity, residual flux density, and hence squareness ratios.
- Extended life tests up to 50,000 tape passes under fairly severe geometric and environmental conditions. These tests included measurement of starting and running torques, debris accumulation, and head wear.

Test procedures and results obtained are outlined in Section 5.3.



Table 11

## DESCRIPTION OF SAMPLE TAPES

Test Number	Binder		Coating Additives			Substrate M=Mylar K=Kapton
	Description	Designation	Oxide Loading (%)	Carbon (%)	Lubricant (%)	
1	Polyamide Modified Aromatic Epoxide	EPON 669 (Versamid 115)	70	—	—	M
2	Polyamide	Elvamide 8061	70	—	—	M
3	Polycarbonate-Amide	AC-26	70	3	—	M
4	Polyamide Modified Aromatic Epoxide	DER 669 (Versamid 115)	70	3	—	M
5	Polyamide	Elvamide 8061	70	3	—	K
6	Polyamide Modified Aromatic Epoxide	EPON 669 (Versamid 115)	70	3	5 <sup>*</sup>	M
7	Polyamide Modified Aromatic Epoxide	EPON 669 (Versamid 115)	80	—	—	M

\*Lubricant: Decafluorobiphenyl

### 5.3 Results and Observations

In order to facilitate comparison between the various tape samples, a matrix was constructed from which specific differences can be identified between the tape samples.

COMPARATIVE MATRIX OF TAPE SAMPLES

	1				
Tape					
2	Change in Polymer				
		2			
3					
			3		
4	Addition of Carbon		Change in Polymer		
				4	
5		Addition of Carbon	Change in Polymer	Change in Polymer	
					5
6	Addition of Carbon and Lubricant			Addition of Lubricant	
					6
7	Change in Oxide Loading			Change in Oxide Loading & Carbon	Change in Oxide Loading, Carbon & Lubricant

For example, comparison of the results obtained from tape samples 1 and 4 will indicate the effect of the addition of carbon, etc.

### 5.3.1 Guideline Tests

The tape samples were subjected to a series of guideline tests which were formulated under NASA Contract No. NAS5-11622, to assist in the selection of magnetic tape in satellite recorder systems. The procedures of these tests are documented in Volume I of the final report of IITRI Project E6134 (NAS5-11622).

The results of these tests are shown in Table 12. It should be noted at this time that none of the tape samples fulfilled all of the guideline tests. These tests, however, were formulated around measurements made on standard instrumentation tapes using more conventional polymer binders. For this reason, the guideline tests may not be directly applicable to the sample tapes fabricated during the program. The tests were undertaken however to act as an initial datum point and, if necessary, will be restructured at a later date in this program.

All of the sample tape types except the polycarbonate-amide successfully satisfied the thermal stability test. The polycarbonate-amide exhibited partial adhesion, more detailed explanation of these results may be obtained with reference to the thermo-adhesive properties of the tape samples in Section 4.4.3 of this report. Several of the tape samples exhibited a weight loss when subject to a benzene soak for 24 hours. This weight loss is however not indicative of the lubricant content of the tape. It should be observed that the losses observed occurred on one binder formulation only, that is the polyamide modified aromatic epoxide. This may be due to an insufficient curing period and will be investigated more fully at a later date.

The resistivity of the oxide/binder surface varied as expected. The addition of carbon to the coating reduced the resistivity (compare tape no. 1 with tape no. 4) whereas an increase of oxide loading (tape 1 and tape 7) increased the resistivity by an order of magnitude. The high value measured

Table 12

## GUIDELINE TESTS

<u>Tape Type</u>	<u>Thermal Stability</u>	<u>Benzene Soak</u>	<u>Resistivity</u>	<u>Chlorine Content</u>	<u>Oxide Dispersion</u>	<u>Flexibility</u>
1	No Effect	0.53%	$5.5 \times 10^{12}$	Negative	46 db	22°
2	No Effect	0.0 %	$9.5 \times 10^{10}$	Negative	32 db	Twist
3	Partial Adhesion	0.0 %	$4.0 \times 10^{10}$	Negative	—	Twist
4	No Effect	0.20%	$2.0 \times 10^{11}$	Negative	42 db	Twist
5	No Effect	—	$1.3 \times 10^{11}$	Negative	45 db	63°*
6	No Effect	0.92%	$1.2 \times 10^{14}$	Negative	32 db	25°
7	No Effect	0.91%	$4.0 \times 10^{13}$	Negative	47 db	20°

\*Kapton base

on tape no. 6 is probably due to a migration of the lubricant (a good dielectric) to the surface of the tape.

As expected, none of the tape samples exhibited a positive indication of the presence of chlorine.

The oxide dispersion measurements indicate that fairly good dispersion was obtained on these initial samples. These results however may be influenced by the overall surface roughness of the tape samples and more so by the lack of any tailoring of the magnetic properties of the tape. Again these results will act as a base line for further comparative analysis.

In most instances the sample tape proved to be slightly stiffer than conventional instrumentation tapes. The exception was tape no. 5 where the substrate used was Kapton. This additional stiffness is not substantially different from the norm and is probably due to residual stresses incurred during the coating process.

### 5.3.2 Friction Test with Various Head Materials

Coefficient of friction measurements were conducted on an Instron testing machine at a speed of one inch per minute. The apparatus used for this measurement is shown in Figure as described in Section 4.3.6. The apparatus was however modified to incorporate dummy magnetic heads of various common head materials instead of the usual slides. Head materials investigated included aluminum, Havar, Alfesil, and combination of these. The results are shown in Table 13.

The control tape, which was included for comparative value only, is a fine polished instrumentation tape. The results indicate that somewhat typical frictional coefficients were obtained from the sample tapes, lower measured values on the harder materials such as Alfesil were expected. The addition of carbon to the coating appears to increase the frictional value (tapes 1 and 4) the further addition of lubricant then lowers the values as expected (tapes 4 and 6).

Table 13  
COEFFICIENT OF FRICTION ON VARIOUS HEAD MATERIALS

<u>Test Number</u>	<u>Head Material</u>				
	<u>Aluminum</u>	<u>Alfesil</u>	<u>Havar</u>	<u>Aluminum- Alfenol</u>	<u>Havar- Alfenol</u>
1	.219	.204	.216	.305	.203
2	.261	.187	.225	.241	.248
3	.336	.233	.267	.375	.289
4	.450	.264	.325	—	—
5	.329	.229	.325	—	—
6	.316	.221	.256	—	—
7	.499	.392	.372	—	—
Control	.304	.306	.337	.298	.283

### 5.3.3 Magnetic Properties

The magnetic properties of the magnetic tapes produced for evaluation were in no way tailored for optimum performance. It was, however, considered necessary to measure these properties in order to form a base for further comparison at a later date. The magnetic properties were measured using a 60 cps dynamic B-H hysteresis loop testing calibrated to known standards. The results are shown in Table 14. For comparison two control tapes were included, one is a representable acicular gamma ferric oxide magnetic tape currently available, and the other a cobalt modified tape structured to exhibit a higher coercivity and superior short wavelength performance.

Comparison of the value of coercivities, i.e., the field strength where the intrinsic flux density is zero, reveals good agreement for all of the tapes as would be expected from the use of acicular gamma ferric oxide as the magnetic material. The residual flux density, i.e., the flux density at which the magnetizing field strength is zero, does show some variability between tape samples. This measurement is somewhat indicative of the anticipated output at short wavelengths and the variability at this stage is probably due to inadequate dispersion and associated coating abnormalities. The residual flux however, i.e., the product of the residual flux density and cross sectional area is fairly consistent for all samples. The residual flux is indicative of output anticipated at long wavelengths and it is directly related to oxide thickness and percent oxide loading. It should be noted that average values compared favorably with Control B (superior short wavelength response) while tape no. 7 (higher overall loading) was more typical of standard tapes where 1.0 maxwell is a nominal value for 1/2 inch wide tape.

The saturation flux density was measured to allow the evaluation of the squareness ratio. This ratio is an indication

Table 14  
COMPARISON OF MAGNETIC PROPERTIES

<u>Tape</u>	<u>Coercivity (oersteds)</u>	<u>Residual Flux Density (gauss)</u>	<u>Residual Flux (maxwells)</u>	<u>Saturation Flux Density (gauss)</u>	<u>Squareness Ratio</u>
1	270	720	0.40	1135	0.64
2	285	475	0.45	765	0.62
3	295	990	0.37	1500	0.66
4	270	700	0.72	1180	0.60
5	270	1075	0.43	1790	0.60
6	275	560	0.45	970	0.57
7	260	770	1.02	1280	0.60
Control A	265	920	0.90	1200	0.77
Control B	450	1320	0.47	1470	0.90



of the degree of orientation of the acicular particles and varies from 0.5 for randomly orientation to a theoretical 1.0 for complete orientation, a typical value in practice is 0.73. No attempt was made during the coating process of these sample tapes to orient the particles, the measured values of approximately 0.63 are therefore indicative of partial orientation obtained during that coating process.

#### 5.3.4 Extended Life Tests

These extended life tests were undertaken to evaluate the endurance of the sample tapes to a large number of tape passes. The tests were carried out on endless loop transports which had been used extensively under Contract No. NAS5-11622. These loop transports were ideally suited to the short length samples and also allowed a large number of tape passes to be compiled in a relatively short time period.

The endless loop tape transports were designed to have an essentially square tape path with the oxide on the outside. A single capstan drive was utilized. This capstan was belt coupled to a DC servo-controlled capstan motor. One of the idlers was mounted on a counter balanced arm appropriately weighted to provide tape tension. Crowned tape guidance was used, and roller diameters were two inches or greater to minimize tape stresses. The DC motor, generator, and speed control was Motomatic Model E-550, built by Electrocraft Corporation, Hopkins, Minnesota. The motor generator has two windings on the same armature, one for driving the motor and the other for generating a voltage proportional to speed. This voltage is then compared to a reference set by the speed control adjustment thereby maintaining constant speed throughout these tests. Two of the endless loop transports were run parallel in an environmental chamber (Tenney Model TH-10) capable of controlling both temperature and relative humidity.

The conditions selected for these extended life tests

were considered to be fairly harsh in an attempt to partially accelerate failure. The selected conditions were:

Temperature	45°C
Humidity	15% RH
Tape Speed	30 ips (high), 1.0 ips (low)
Direction	Bidirectional
Tape Tension	12 oz (1/2 inch tape)
Wrap Angle (total)	30°
Head Material	Aluminum
Number of Passes	50,000

The parameters monitored during these extended life tests included starting and running torques, debris formation and head wear.

#### 5.3.4.1 Starting and Running Torques

Two measurements, starting and running torque, were made on the loop transports. These measurements were obtained from the DC capstan motor current. In monitoring starting torque, the speed adjustment was first set for the desired speed (usually 30 ips), with the torque (motor current) limiter to zero. The torque limiter was then slowly increased until tape motion began. The calibration of the torque readings was accomplished by running tape on the transport out of contact with the head. In this manner, losses due to the motor brushes and bearing friction could be subtracted. The torque was expressed in terms of capstan torque, i.e., motor torque times a 5.89 ratio of speed reduction.

The measurement of both starting and running torque was made every 1000 tape passes and the resulting values for each tape plotted against number of tape passes up to 50,000. In order to facilitate comparison of the results obtained during these tests, use has been made of the comparative matrix shown on page 57 and the graphs of starting and running torque displayed accordingly.

Figure 10 displays the difference between the polyamide modified aromatic epoxide (1) and polyamide binder (2). It should be noted that the running torques are very similar up to 50,000 tape passes centered around a level of 5.0 oz-in. in tape no. 2 the polyamide has superior starting torque values in that these appear slightly lower than tape no. 1. Neither tape type exhibited a dramatic increase in either of the measured torque values up to 50,000 tape passes.

Figure 11 illustrates the effect of the addition of carbon to the binder mix for the epoxide tape. Starting torques are substantially reduced to a level of 2.5 oz-in. Running torque values are also reduced but to a lesser extent. Again no substantial increase in either torque measurement was noted.

Figure 12 illustrates the effect of the addition of carbon and lubricant to the basic polymer for the epoxide tape. Running torques are substantially reduced, but the effect on the starting torque is not so apparent. It should also be indicated that the lubricated tape (6) shows a tendency towards a gradual increases up to 50,000 tape passes.

Figure 13 illustrates the effect of adding a lubricant (tape 6) to a carbon/polymer coating. It should be observed that although the running torque is reduced, an increase in the starting torque was experienced. This result is indicative of the importance of tailoring the lubricant content of the tape, and this area will be studied in more detail as the coatings are optimized during Phase II of this program.

Figure 14 illustrates the difference in running and starting torque for a change in the oxide loading of the epoxide binder. It is considered however that the curves are, at first sight, misleading. Although the higher oxide loading (tape 7) appears to exhibit lower values for both running and starting torques the important parameter to observe is the rate of change during the initial 15,000 tape passes. Visual

observations made during this test indicated that severe oxide shedding occurred around 20,000 tape passes. It is conjectured that initial damage occurred up to 15,000 tape passes at which a change in the surface properties in an apparent lowering of the starting and running torque values.

Figure 15 illustrates the differences observed with the addition of carbon to the polyamide. As expected, reductions in both the running and starting torques were observed. Figure 16 shows the differences between the polyamide and epoxide coatings both with carbon. It can be seen that the measured starting torques are very similar although differences were observed between the running torques, the polyamide exhibiting a lower value.

Figure 17 indicates the difference observed between the polycarbonate-amide (tape 3) with that of the epoxide (tape 4), both with the addition of carbon. Although the polycarbonate-amide exhibited lower running values of torque and slightly higher starting values of torque than the epoxide tape, severe oxide shedding occurred and the test on the polycarbonate-amide was terminated at 15,000 tape passes. Figure 18 illustrates the difference between the polycarbonate-amide and the polyamide tape samples, again both with a carbon additive. What should be observed is although the magnitude of running and starting torques are very similar they bear little relationship to the tenacity or integrity of the coatings, that is the ability of the coating to withstand a large number of tape passes without shedding.

The last of these curves, Figure 19, illustrates the differences observed for a change of head material only. Two head materials, namely chrome and aluminum, were examined using an epoxide coating with carbon (tape no. 4). Little difference between the absolute values of both starting and running torques were observed, however substantial differences

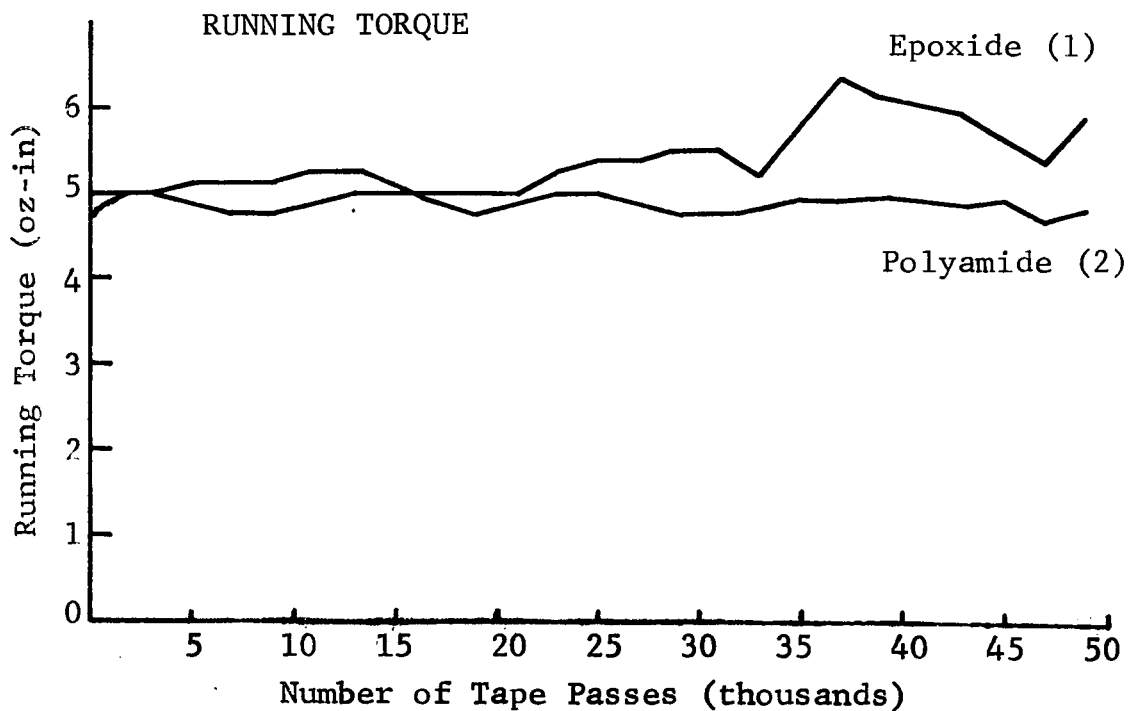
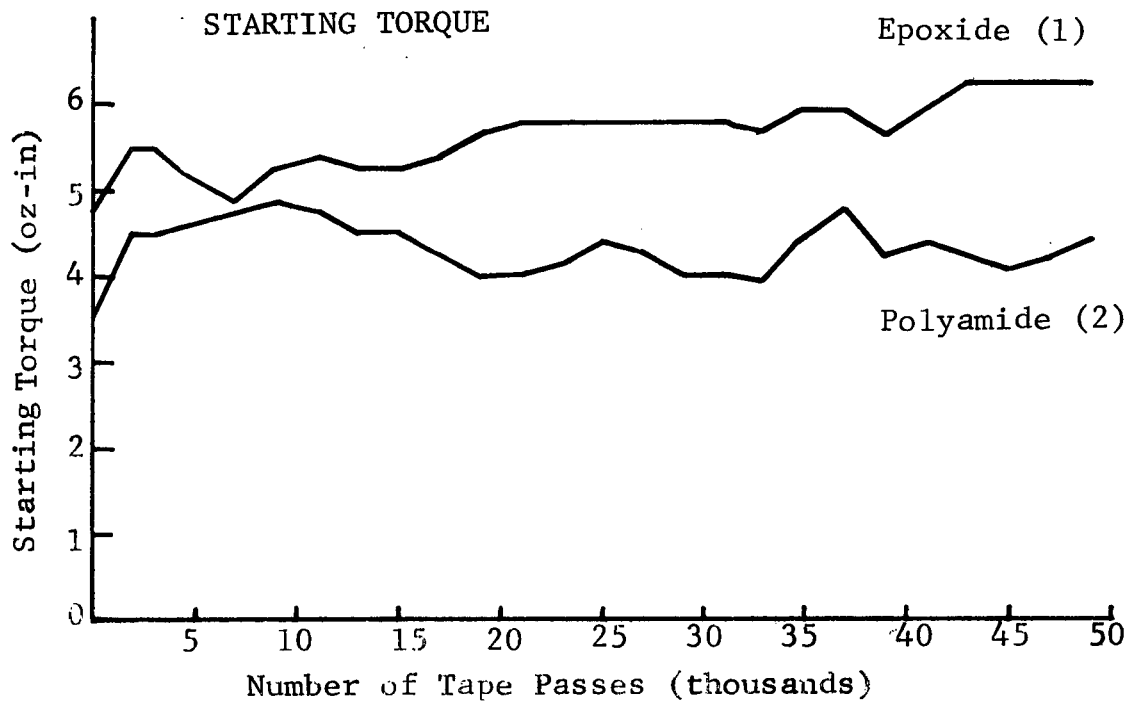


Fig. 10 EXTENDED LIFE TESTS - RUNNING AND STARTING TORQUES,  
COMPARISON OF POLYAMIDE AND EPOXIDE BINDERS

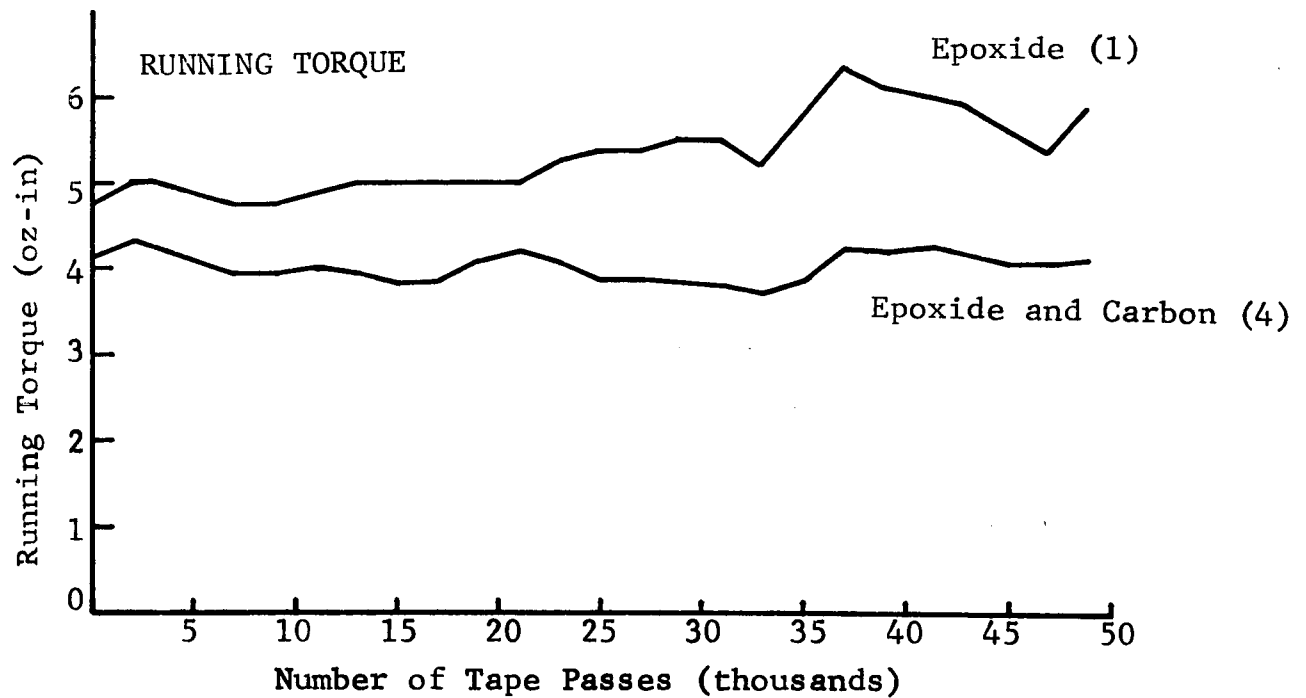
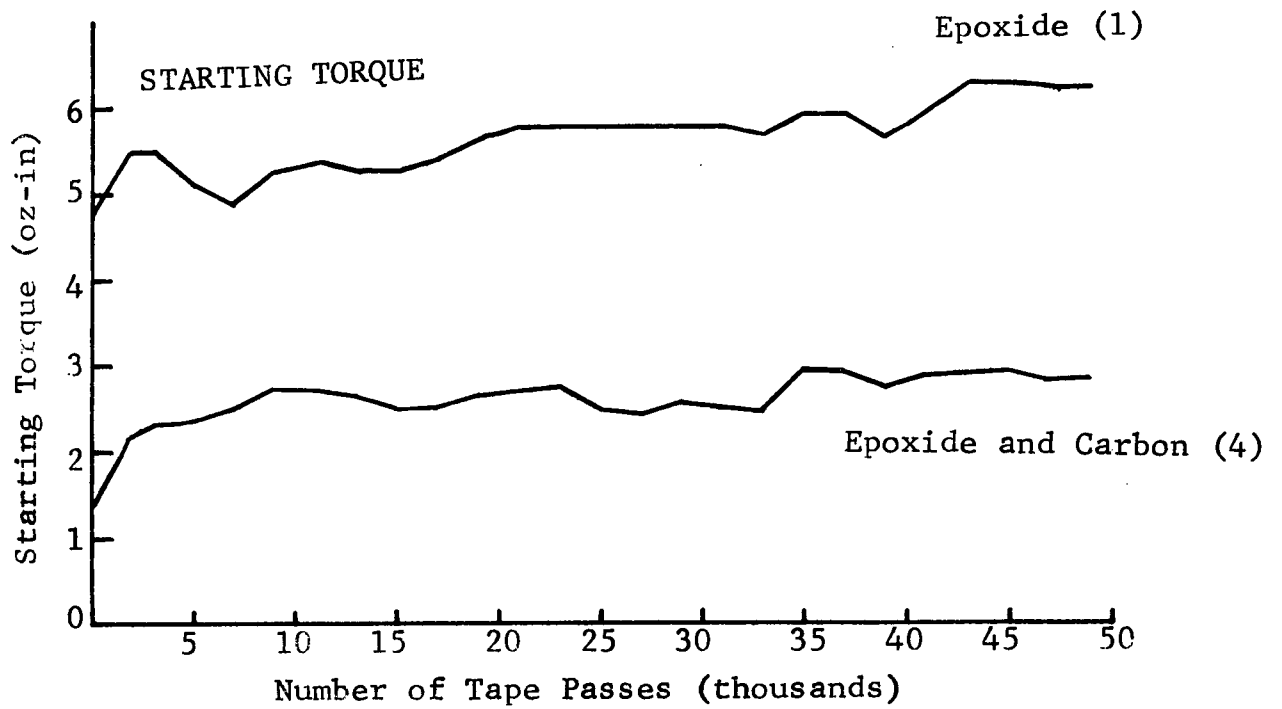


Fig. 11 EXTENDED LIFE TESTS - RUNNING AND STARTING TORQUES,  
COMPARISON OF CARBON ADDITIVE TO EPOXIDE BINDER

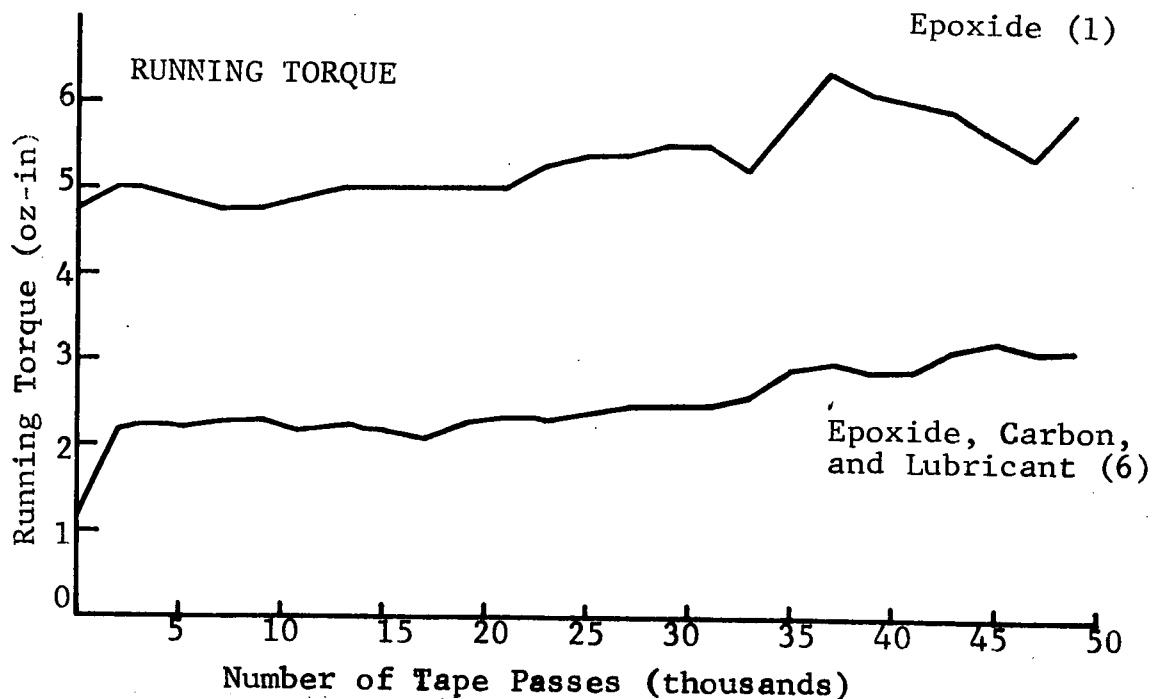
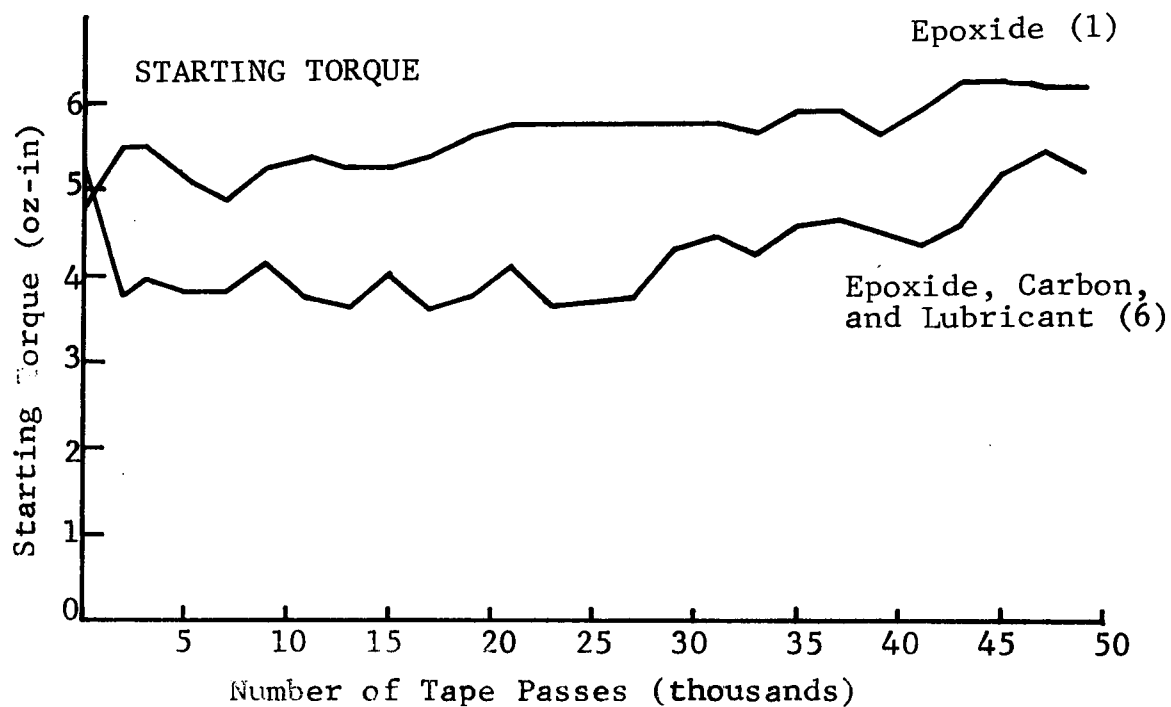


Fig. 12 EXTENDED LIFE TESTS - RUNNING AND STARTING TORQUES,  
COMPARISON OF CARBON AND LUBRICANT ADDITIVE TO  
EPOXIDE BINDER

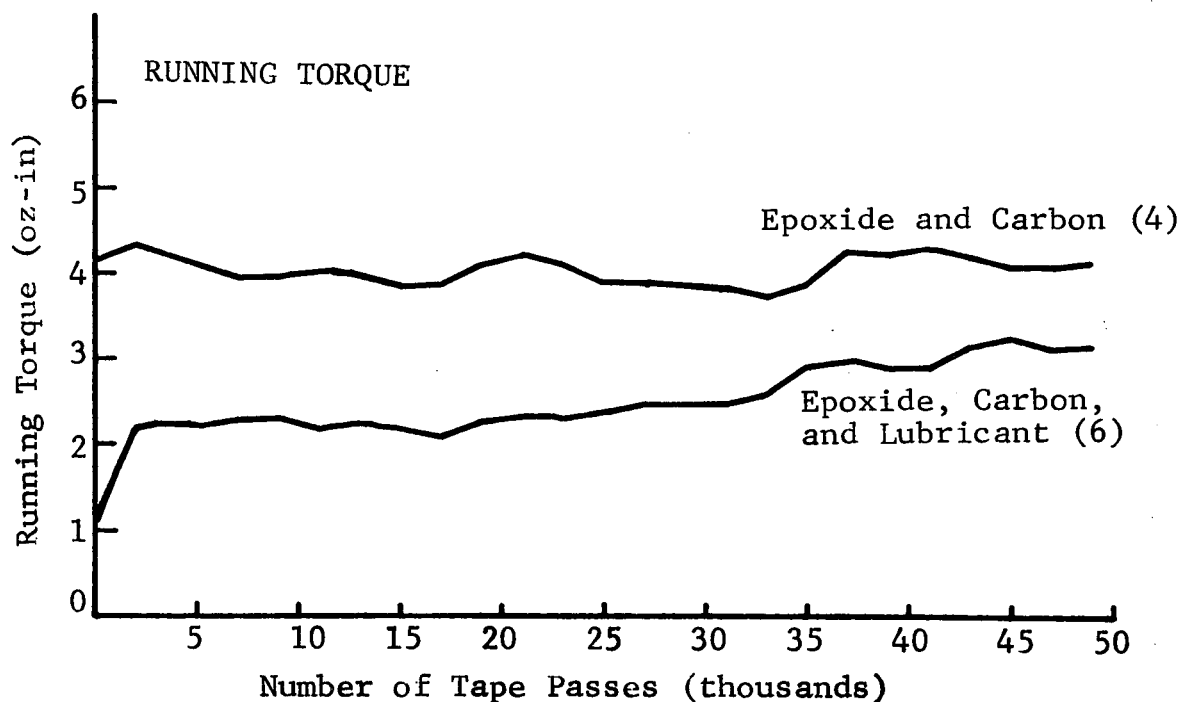
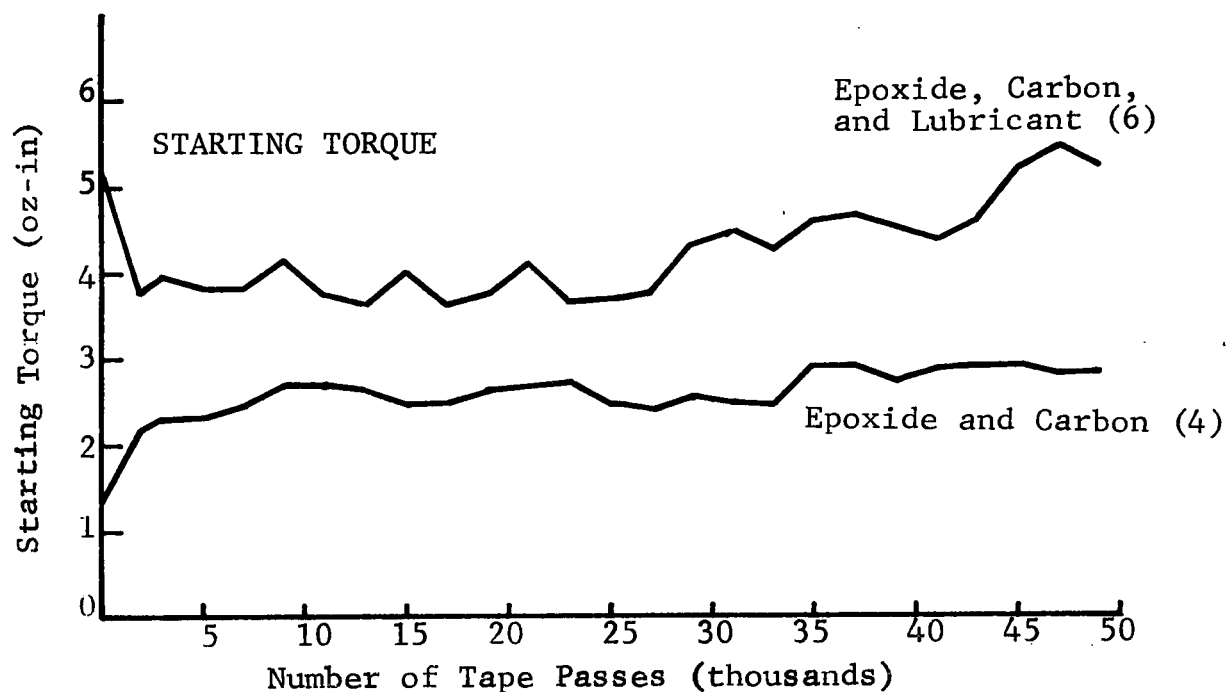


Fig. 13 EXTENDED LIFE TESTS - RUNNING AND STARTING TORQUES,  
COMPARISON OF LUBRICANT ADDITIVE TO EPOXIDE BINDER



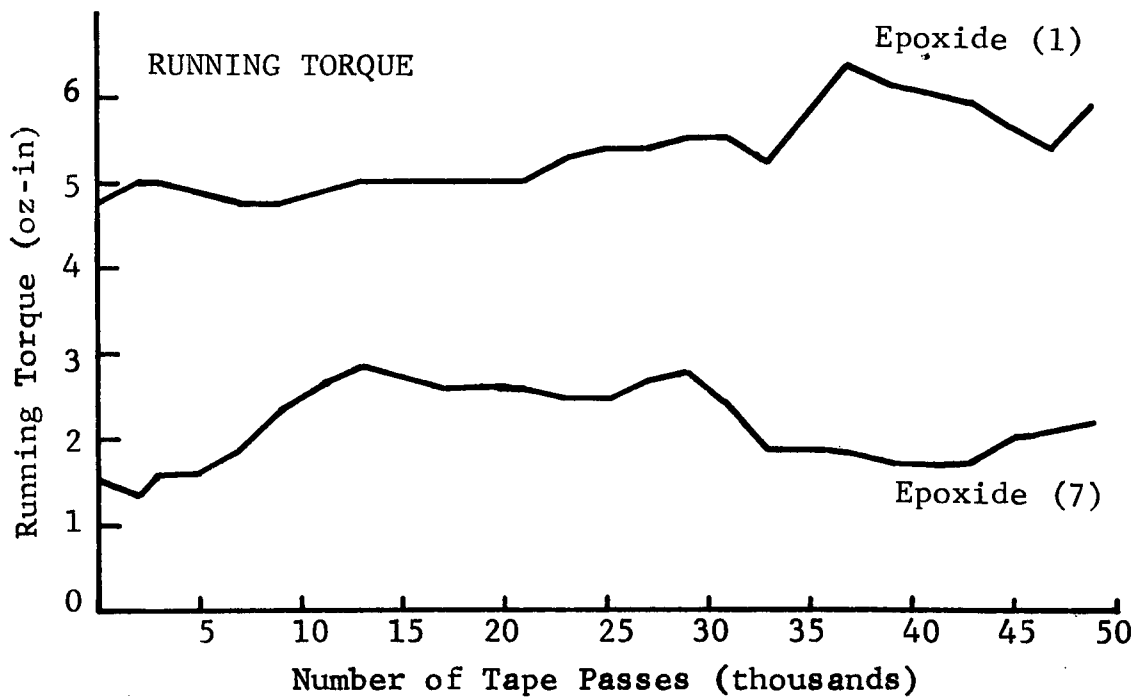
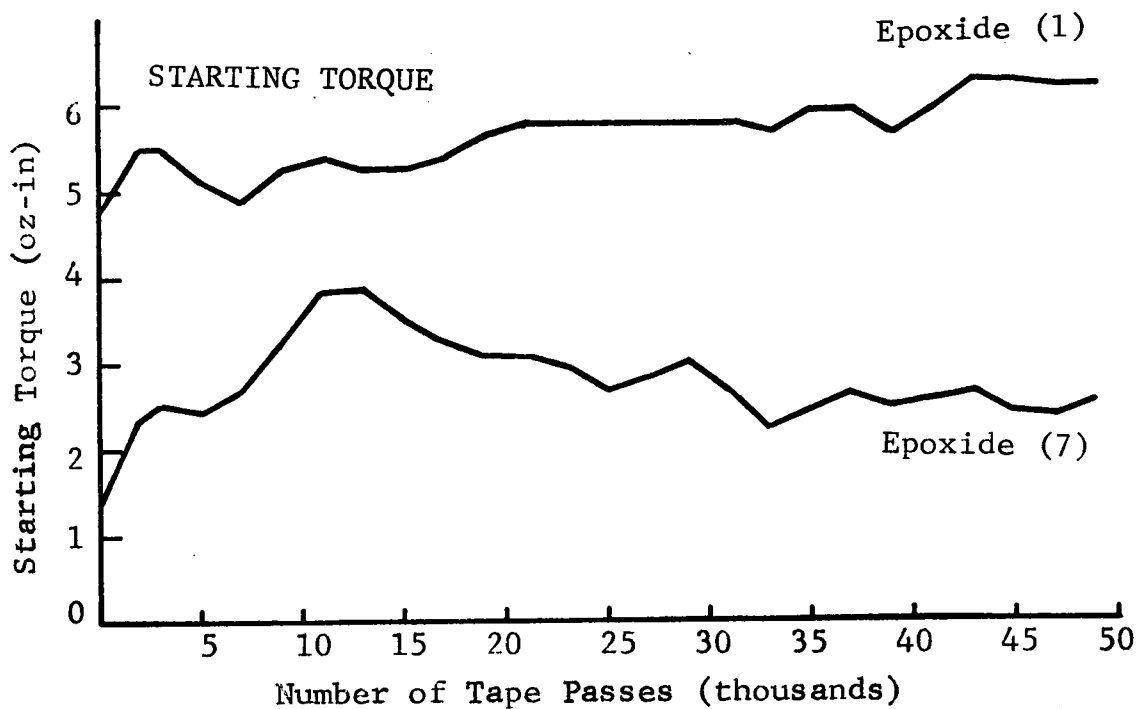


Fig. 14 EXTENDED LIFE TESTS - RUNNING AND STARTING TORQUES,  
COMPARISON OF OXIDE LOADING FOR EPOXIDE BINDER

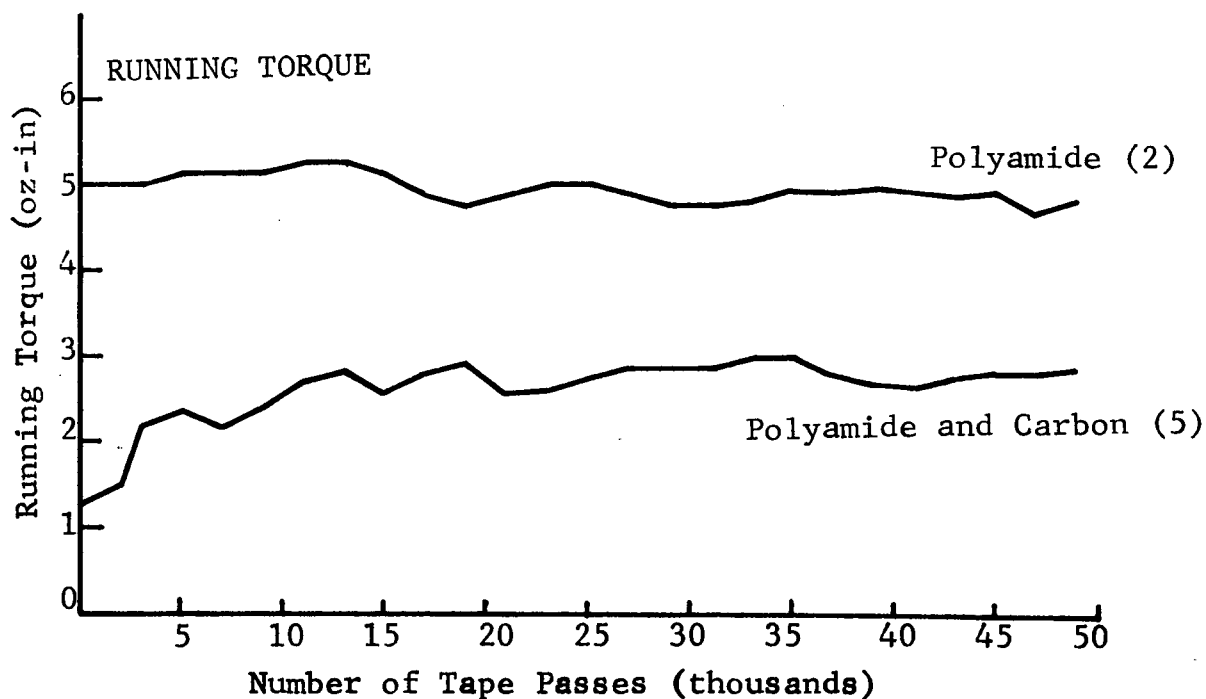
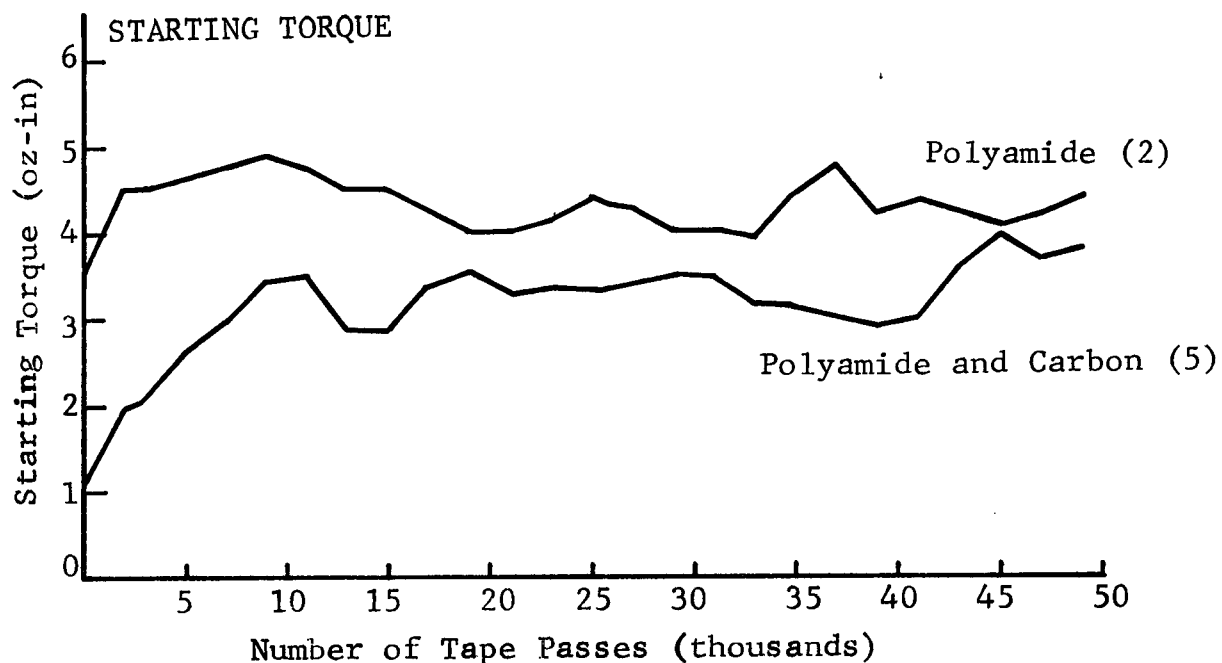


Fig. 15 EXTENDED LIFE TESTS - RUNNING AND STARTING TORQUES, COMPARISON OF CARBON ADDITIVE TO POLYAMIDE BINDER

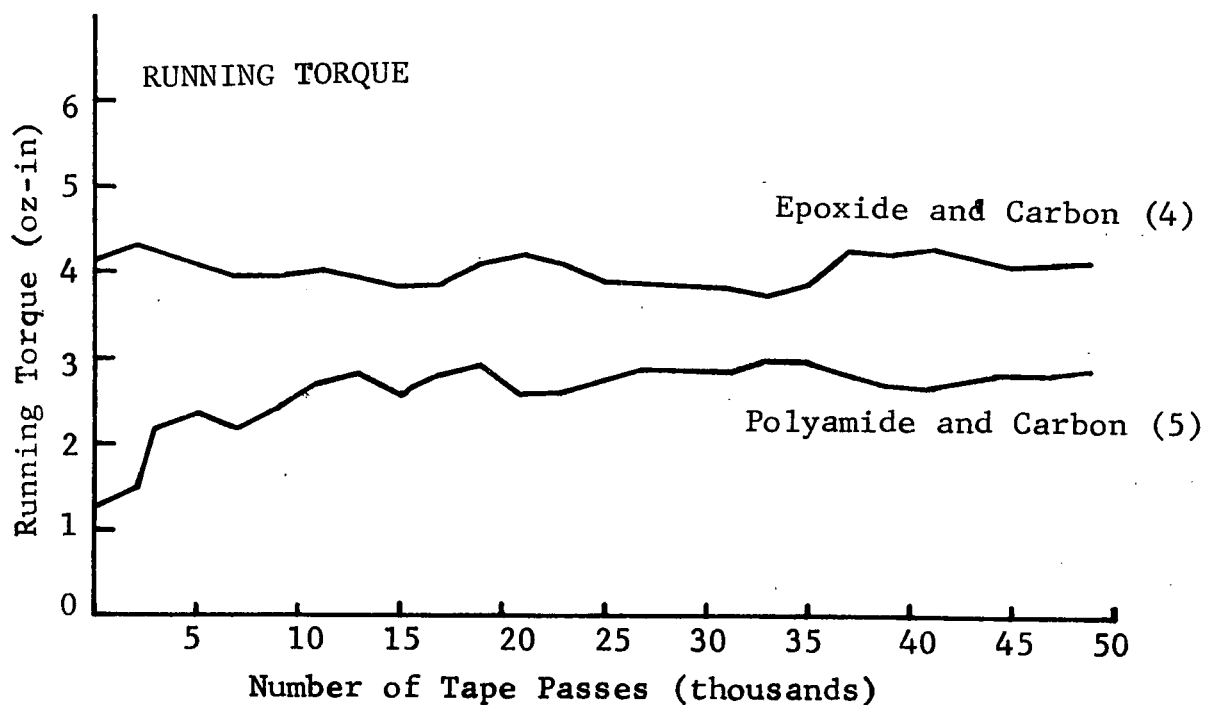
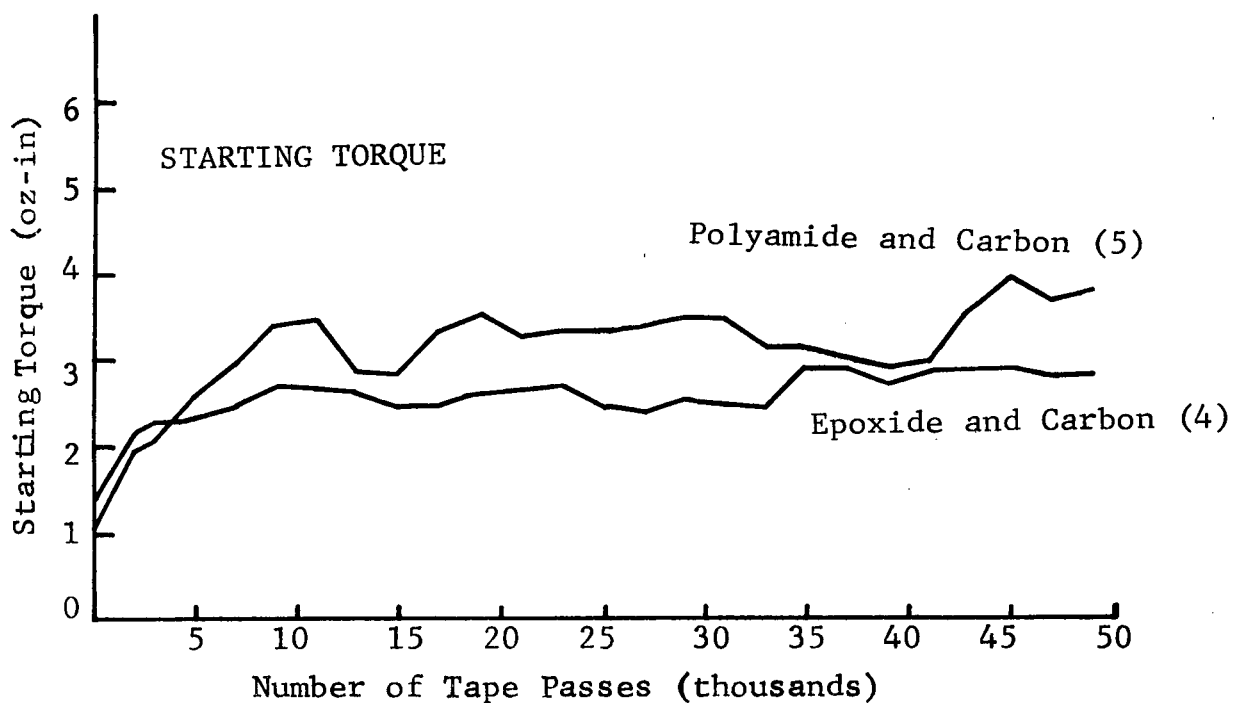


Fig. 16 EXTENDED LIFE TESTS - RUNNING AND STARTING TORQUES, COMPARISON OF CARBON ADDITIVE TO POLYAMIDE AND EPOXIDE BINDERS

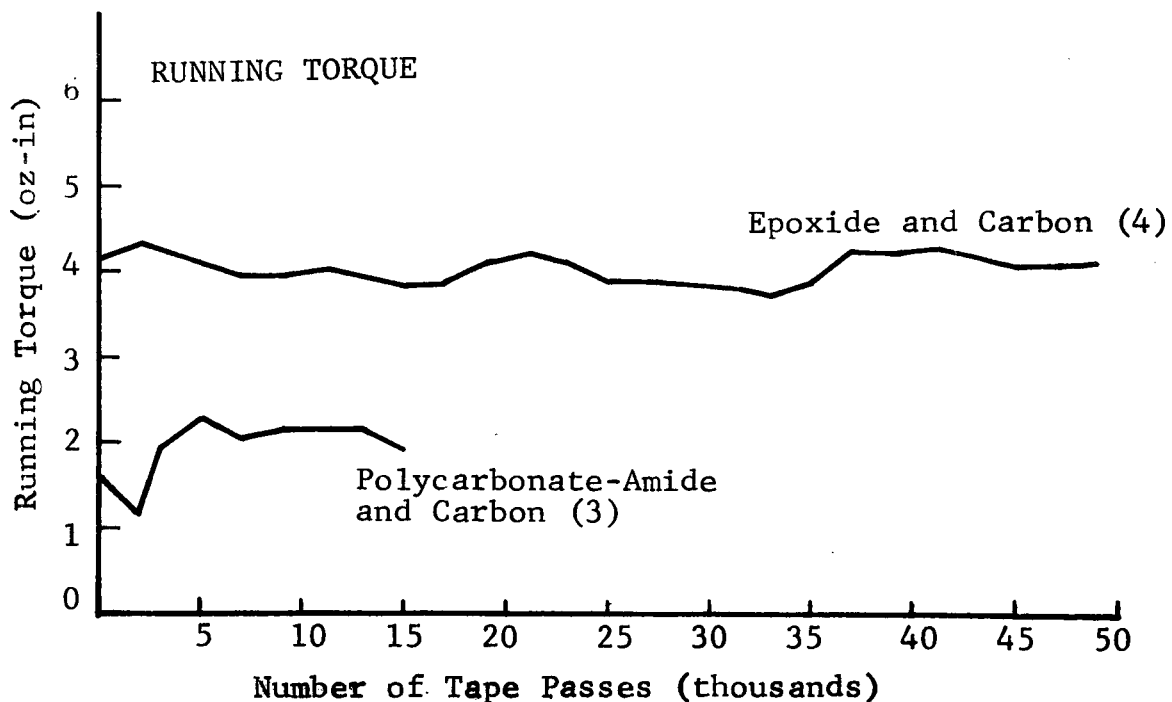
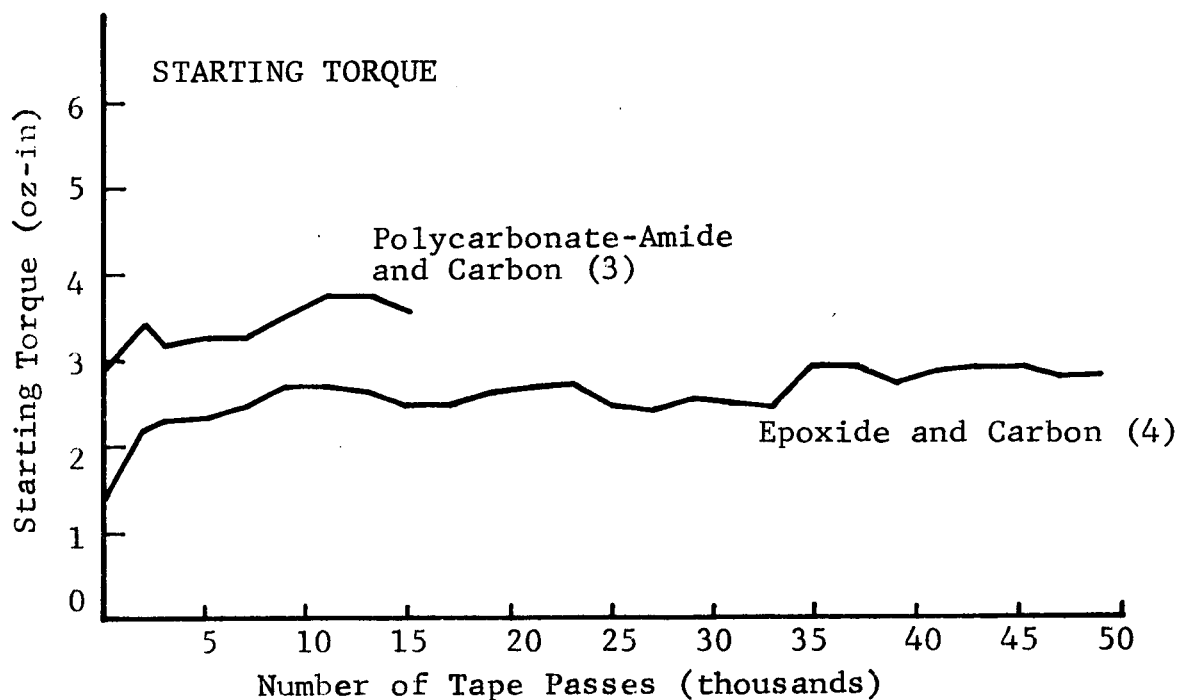


Fig. 17 EXTENDED LIFE TESTS - RUNNING AND STARTING TORQUES, COMPARISON OF CARBON ADDITIVE TO POLYCARBONATE-AMIDE AND EPOXIDE BINDERS

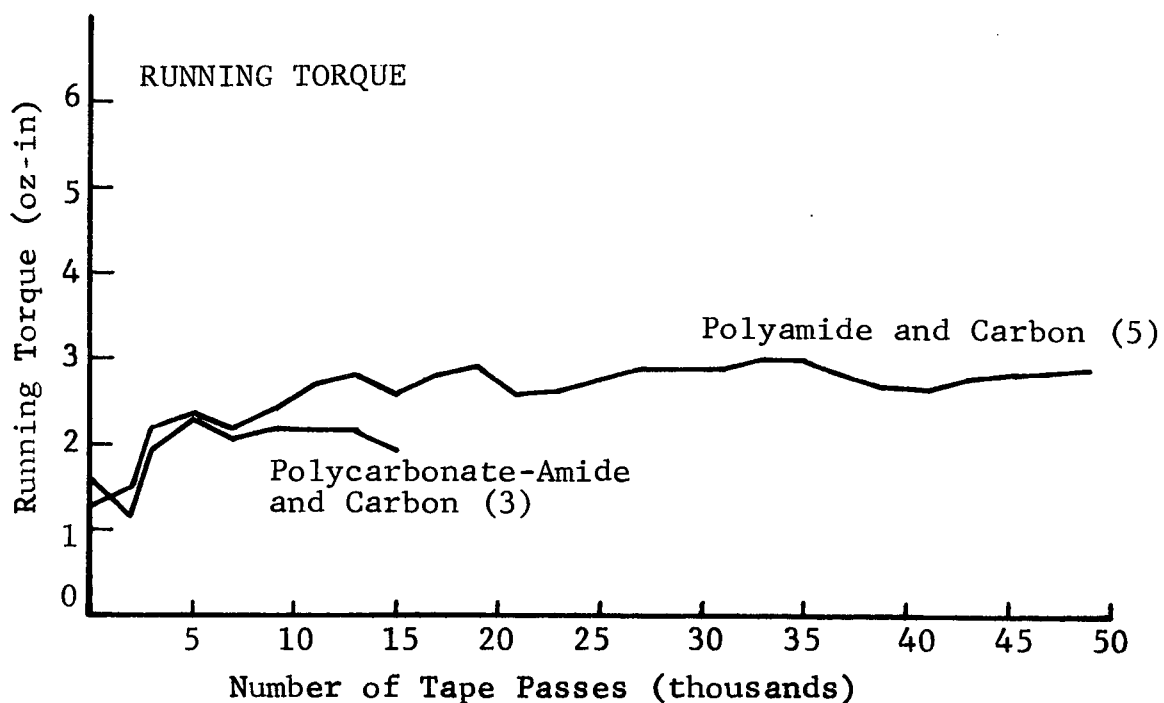
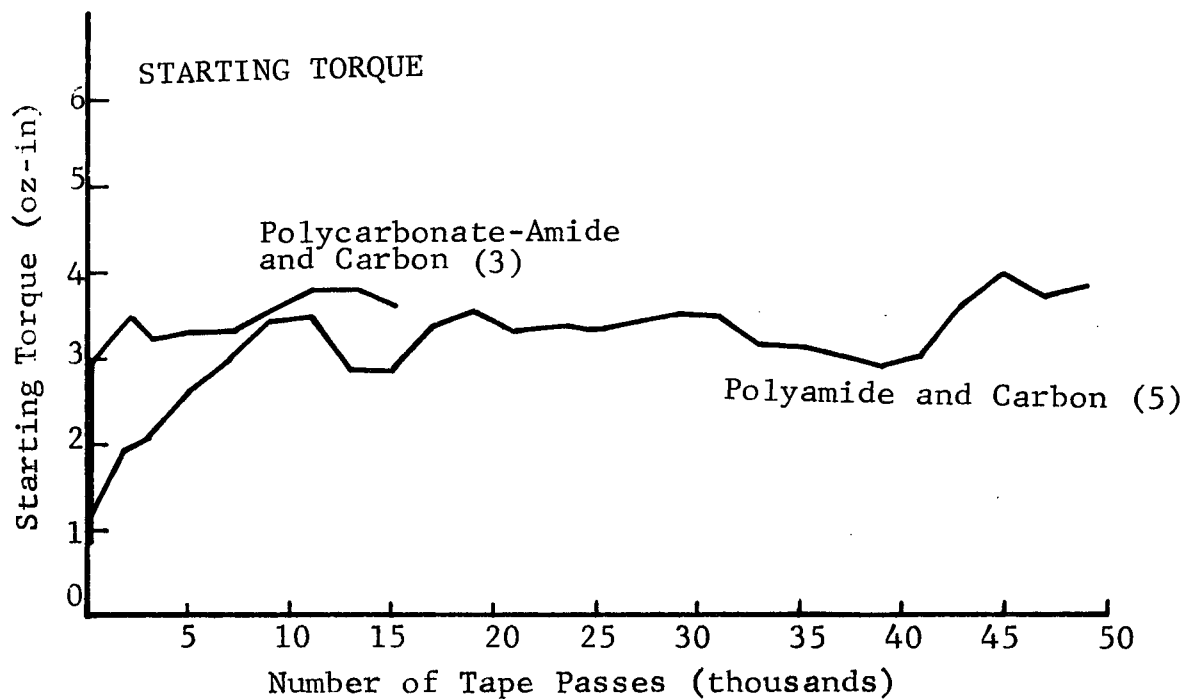


Fig. 18 EXTENDED LIFE TESTS - RUNNING AND STARTING TORQUES,  
COMPARISON OF CARBON ADDITIVE TO POLYCARBONATE-AMIDE  
AND POLYAMIDE BINDERS

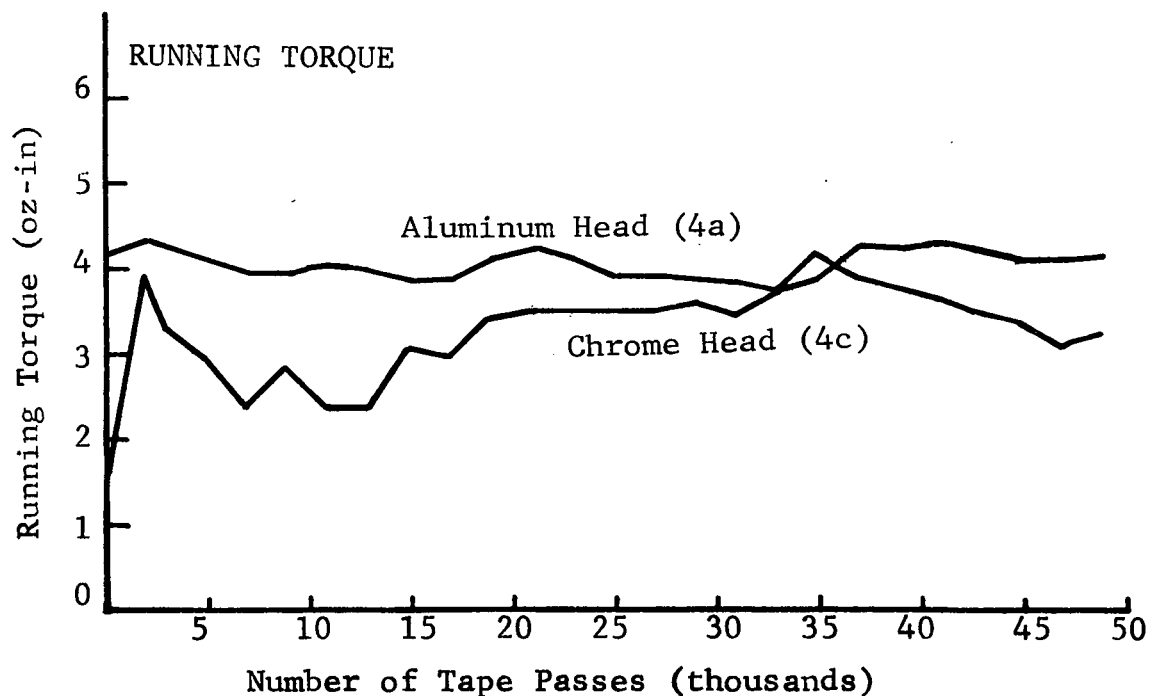
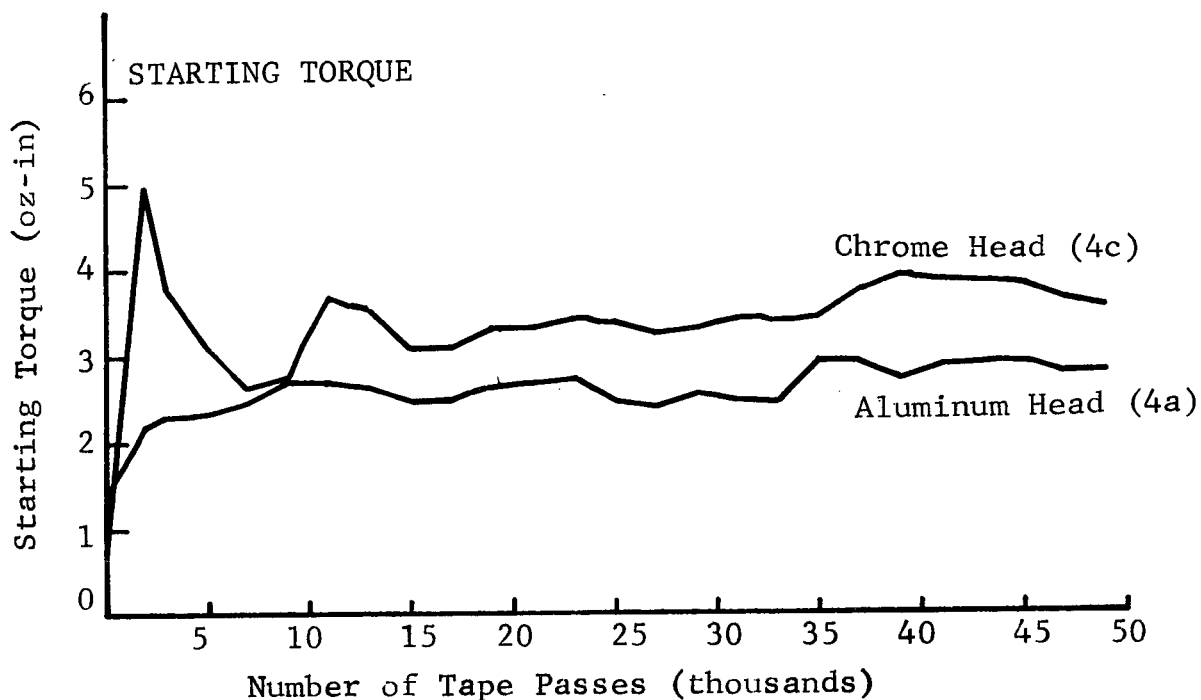


Fig. 19 EXTENDED LIFE TESTS - RUNNING AND STARTING TORQUES, COMPARISON OF HEAD MATERIALS ON CARBON ADDITIVE EPOXIDE BINDER.

in debris formation and head wear were documented and are reported in the following sections.

#### 5.3.4.2 Debris Formation

The formation of oxide binder debris, where this occurred, was observed visually during the extended life tests. At the conclusion of each test the head was carefully removed, weighed, cleaned of any adhering debris, and reweighed. The resulting weight of accumulated debris was therefore indicative of the tape samples tenacity or integrity. The conditions of test, as indicated earlier were severe, but these conditions were maintained constant for each tape sample and are therefore worthy of comparison. The results are shown in Table 15.

Table 15

#### DEBRIS ACCUMULATION

<u>Tape Sample</u>	<u>Weight of Debris (μgm)</u>	<u>Comments</u>
1	>1400	Some Debris Lost
2	>1300	Some Debris Lost
3	5300	17,000 Passes Only
4	110	—
4 repeat	0	Chrome Head
5	1300	—
6	150	—
7	>4300	Some Debris Lost

It is immediately evident that the severe debris forming tapes were those that contained an oxide/binder mix only, with no other additives (tapes 1, 2, and 7). In each case the amount of debris was excessive and some was lost to the floor of the test chamber. The addition of carbon to the epoxide tape (tape 4) dramatically inhibited the debris formation, this was further reduced when the head material was changed to

chrome from that of aluminum. The further addition of a lubricant (tape 6) did not appear to influence the amount of debris observed. In the case of the polyamide tapes, the addition of carbon (tape 5) reduced debris formation, but not to the same extent as that of the epoxide based tapes.

#### 5.3.4.3 Abrasivity

The abrasivity of the sample tapes was identified by measuring the amount of wear sustained by the dummy heads over 50,000 tape passes. The results are shown in Table 16.

Table 16

#### AVERAGE HEAD WEAR (50,000 TAPE PASSES)

<u>Tape Sample</u>	<u>Average Head Wear (<math>\mu</math> in)</u>	<u>Comments</u>
1	>1000	
2	>3200	
3	1600	17,000 Passes Only
4	1650	
4	450	Chrome Head Material
5	1250	
6	1050	
7	>4200	

As expected by the harsh nature of this test, the wear rates sustained by the dummy heads are high. No attempt was made to minimize head wear by reducing operating tensions and tape wrap angles as usually recommended. The results show good correlation between debris formation and head wear. Tapes consisting of an oxide/binder surface only (tapes 1, 2, and 7) exhibited excessive wear, while the addition of carbon (tapes 3, 4, and 5) minimized the head wear rate. The difference between the hard chrome head and the softer aluminum head structure is also



evident when used on an epoxide based tape (tape 4). Although these results are excessive, probably due to the lack of surface finish treatment of the preliminary tapes, they do form a base for further comparison as the optimization of the coatings proceed during Phase II of this program.

## 6. SUMMARY AND RECOMMENDATIONS

The intent of Phase I of this four phase program was the identification of four generically different polymer binders capable of fulfilling the requirements necessary to become desirable candidates for a five year magnetic tape. The program was therefore structured around a detailed investigation of a variety of polymer groups. This detailed form of analysis, undertaken during this phase, allowed the properties of these materials to be accurately determined by fabricating free films of these polymer groups independent of the carrier base film. The ability to form free films of such polymers and their combinations with various oxide loadings and other necessary additives was instrumental in allowing a rapid and natural progression to the selection of four candidate polymers. These are: a polyamide modified epoxide, a polyamide, a polycarbonate-amide, and a polyimide.

Once established, these candidate polymers were extensively analyzed to determine their physical, mechanical, and chemical properties; specific attention was paid to their behavior in areas that are known to be life limiting, such as frictional properties and thermo adhesive properties.

Following this detailed analysis, short length tape samples were produced in pilot quantities. These samples were then further evaluated in the laboratory under actual running conditions. These evaluation tests were specifically oriented towards determining quantitized data which would form a base-line of information to be used as the program progressed into the optimization phase. Particular attention was directed towards the physical and magnetic recording properties of the tape samples. Frictional properties with head materials currently in use were measured, as were the intrinsic magnetic properties of the tape samples. Extensive life tests were performed up to 50,000 tape passes at fairly severe running

conditions to establish any adverse rate of change of running and starting torques that may be experienced. At the same time, base line information in debris formation and head wear was collected.

The primary objectives of the first phase of this program were successfully accomplished in that specific tape performance criteria were established for a five year magnetic tape and four generically different polymer candidates were identified. During the course of this Phase I effort pilot quantities of short length tape samples were also produced and extensively evaluated. Although no attempt was made to optimize these coating systems at this stage, the evaluatory results in many areas proved impressive. Investigations carried out under NASA Contract NAS5-11622 indicated that many currently available tape types exhibited a failure mode where either the starting or running drag of the system increased dramatically and catastrophically during the initial 10,000 tape passes. The results obtained during the evaluation of the short length tape samples proved extremely encouraging, in that most of the sample tapes examined exhibited stable low level torque values under harsh conditions up to 50,000 tape passes. The success of these preliminary tape samples to withstand these severe conditions for so many bidirectional tape passes indicates that the preliminary analyses and selection criteria established during this program are sound, and that the formulation of a five year magnetic tape with properties selected specifically for unattended operation in spacecraft tape recorders is well under way.

The next step in attaining this goal of a five year magnetic tape is the optimization of the coatings system. This will be undertaken during Phase II of this program and will extend over the next 12 months. During this time the four developed polymers will be optimized in terms of their reaction conditions, molecular weight relationships, coating stability

and affinity for pigments and additives. This will be followed by the adjustment of the resin-pigment proportions, the selection and concentration of the dispersion promoting agents and lubricating agents and optimization of the tape coating procedure. Throughout this effort continued testing and evaluation will be carried out in order to critically assess the coatings behavior in relation to the physical and chemical properties of the binder. At the same time, continued testing of the tapes performance will be undertaken, this will include endurance life testing, frictional behavior, susceptibility to debris formation, and abrasivity. At the conclusion of these extensive Phase II efforts the two most promising magnetic coatings will be selected from the four generically different polymers currently identified, and these coatings will be carried into the next refinement phase.

In conclusion, we feel that the work carried out and the results achieved during the initial phase of this program indicate that definite progress has been made towards a five year magnetic tape. Hence, the aim towards which this program is directed will be successful in accomplishing the fabrication and delivery of a magnetic tape designed specifically for long term use in attended spacecraft tape recorders.

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